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## CAKE AND CAKE MAKING INGREDIENTS[1]

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### Introduction

Cake is a product made with innumerable combinations of ingredients, flour being the basic component. Although flour has been used mainly for bread making, especially in the wheat-consuming countries, since time immemorial, every nation has been sufficiently artful, inventive, and technical to combine certain other ingredients with flour and from such a mixture prepare cake, a product of greater delicacy and tenderness than bread.

### History of Cake Making

The origin of cake is shrouded in mystery and lost in antiquity. Jeremiah, the Jewish prophet records, to be sure, that "the women knead their dough to make cakes." This refers really to some form of "pancakes" and not to cake as understood today. Shakespeare, however, had real cake in mind—"Your cake here is warm within" (A Comedy of Errors), and "Our cake's dough on both sides" (The Taming of the Shrew), refer no doubt to what we know as cake. In more modern times Marie Antoinette attained much of her fame by her suggestion that the poor people, who were clamoring for bread, should eat cake instead. Now-a-days, cake characteristic of each nation is to be found. Thus we speak of German cake (*kuchen*), French cake (*gateau*), English cakes, etc.

The following recipe was published in *Country Contentments* or *The English Housewife*, by G. M. Loudon, in 1623:

[1] Food Research Division Contribution No. 250.

**To Make Spice Cakes**

"To make excellent spice cakes, take halfe a pecke of very fine wheat-flower, take almost one pound of sweet butter, and some good milke and creame mixt together, set it on the fire, and put in your butter, and a good deale of sugar, and let it melt together; then straine saffron into your milke a good quantity; then take seven or eight spooneful of good ale barm, and eight eggs with two yelkes and mix them together, then put your milke to it when it is somewhat cold, and into your flower put salt, aniseedes bruised, cloves and mace, and a good deale of cinamon; then worke all together good and stiffe, that you need not worke in any flower after; then put in a little rose water cold, then rub it well in the thing you knead it in, and worke it thoroughly; if it be not sweet enough, scrape in a little more sugar, and pull it all in peeces, and hurle in a good quantity of currants, and so worke all together againe, and bake your cake as you see cause in a gentle warme oven."

The cake of that period was thus a mixture of "sugar and spice and everything nice." In the early days there was no baking powder; cake was leavened by means of yeast. The dough was similar to bread dough but was enriched with eggs, sugar, fruit, and spices.

Early American cakes were made with molasses from the West Indies; fruit from Spain and Italy; raisins and currants from the Mediterranean; and spices brought in by the Dutch merchants. The eggs, flour, and butter were of home production. Yeast was used as a leavening agent for cake in America as late as 1819. Somewhat later, pearl-ash (potassium carbonate) was introduced into such products as ginger cakes and molasses cakes. Sour milk was also used, making products tender, soft, and delicious, a welcome departure from the solid cakes of sweetened bread dough that had previously been used. The sour milk and molasses furnished the acids that reacted with the potassium carbonate and produced the carbon dioxide which leavened the cakes. "Pearl-ash" was included in recipes as late as 1835. More recently bicarbonate of soda has replaced the potassium carbonate as a source of carbon dioxide in cake baking. Both sour milk and molasses are frequently used at present in making spice cakes and cookies, but for leavening, other products have largely been replaced by acid salts mixed with bicarbonate of soda and starch to form baking powder, which today is used in most of our baked goods. The history of cake making is correlated with the development of leavening agents from barm to baking powder.

## Importance of Cake Industry

The cake industry of the United States is one of the most important baking industries. More flour is used by the baker for cake making than for any other one article of baked goods except bread. Of the 110 million barrels of flour milled in this country, at least 5.5 million barrels are made into cake by commercial bakers and housewives and by bakers connected with hotels, restaurants, charitable institutions, railroads, steamboats, etc. One barrel of flour will produce more than

800 pounds of cake. According to the Bureau of the Census the value of commercial cake made in this country in 1931 was approximately $171,593,605. No corresponding figures for cake production in other countries are available to the authors.

## Consumption of Cake

The importance of cake as an article of food is indicated by the fact that the total cake production by all agencies in this country is between 4 and 5 billion pounds, equivalent to about 1¼ ounces per person per day. Of the other products made from flour the daily per capita consumption is approximately as follows: biscuits and crackers, ½–¾ ounce; macaroni, ¼ ounce, baking powder biscuits, 1 ounce; bread, somewhat more than 7 ounces.

It is estimated that the commercial baker's share of the total cake output of this country is between 20 and 25%. The popularity of the commercial cake is increasing rapidly, and in time relatively few housewives will find it worth while to bake cake or will be so situated that they can conveniently make it. This is happening in the case of bread. Today considerably less than 30% of the bread consumed is made by the housewife; most of it is made by the commercial baker, and in hotels, restaurants, etc.

At present, however, it is estimated that in cities where the baker makes practically all the bread, he is producing not more than 25% of the cake. On the basis that the baker's share of the cake produced is about one-fourth of the total consumed, the annual value of all cake made in this country, by all agencies, is approximately $700,000,000. To obtain a larger share of this vast business is the baker's aim, and in this he can succeed if he is wise enough to make the kind of cake enjoyed by the housewife and her family.

## Types of Cake

Cakes may be divided into two main classes—(1) those made without shortening, *e.g.*, the so-called foam type of cake; and (2) those made with shortening, such as package cake and pound cake.

Cakes made with shortening may again be subdivided into two classes—(1) those made with a large proportion of leavening agents, such as gold cake, and (2) those made with little or no leavening agent, *e.g.*, pound cake.

In the foam type of cake (angel food cake and sponge cake) the open structure or grain is produced largely by the expansion of air incorporated in the egg white during the process of beating.

In gold or package cake the reaction of the baking powder liberates the carbon dioxide within the batter, and upon the application of heat this gas expands, forming the porous structure of the cake.

Pound cake on the other hand contains large proportions of egg, shortening, sugar, and flour. When these ingredients are properly mixed the emulsion formed entraps a small amount of air, which expands during the earlier stages of the baking process. The small pores account for the more nearly solid structure of the heavy types of cake.

This study of cake making has been confined to the following three types:

(*a*) Angel food cake, which is made with egg whites but contains no shortening or baking powder.

(*b*) Gold cake, which is made with egg yolks and contains a small amount of shortening and a relatively heavy charge of baking powder.

(*c*) Pound cake, made with whole eggs, a large amount of shortening, and only a small amount of baking powder. While good commercial pound cake may be made without any baking powder, the best pound cake made in the laboratory contained a little of the leavening agent.

It has generally been assumed that to make the best angel food cake a very weak flour is required; for the best quality of gold cake a somewhat stronger flour can be used; and for pound cake or the heavier types of cake, a moderately strong flour is best adapted. Further, for cake of all kinds, it is best to use flour milled from soft wheat.

## Composition of Cake

Cake, being made of flour, sugar, eggs, milk, and shortening, is naturally rich in carbohydrates and fat (energy foods) and relatively poor in protein. As a rule, cake contains less than half as much protein as does bread.

The composition of the three types of cake, as made in the Bureau of Chemistry and Soils, is given in Table I. The composition of white bread is included for comparison.

TABLE I

Composition of Experimental Cakes and White Bread

| | Angel food cake | Gold cake | Pound cake | White bread |
|---|---|---|---|---|
| | % | % | % | % |
| Moisture | 28.2 | 19.5 | 19.3 | 35.0 |
| Protein (N×6.25) | 7.8 | 6.6 | 7.1 | 8.7 |
| Fat | 0.4 | 20.1 | 23.8 | 2.1 |
| Ash | 0.7 | 1.2 | 0.9 | 1.3[1] |
| Carbohydrates | 62.8 | 52.5 | 48.9 | 52.9 |
| Calories per pound | 1306 | 1899 | 1929 | 1223 |

[1] Including salt.

The composition of five types of commercial cake and of two kinds of bread, as determined by the American Institute of Baking, is given in Table II.

TABLE II

COMPOSITION OF COMMERCIAL CAKE AND OF TWO TYPES OF BREAD[1]

| | Gold cake[2] | Sponge cake | Silver cake | Chocolate layer cake | Devil's food cake | All-milk bread | Whole wheat bread |
|---|---|---|---|---|---|---|---|
| | % | % | % | % | % | % | % |
| Moisture | 23.62 | 22.00 | 25.04 | 21.72 | 24.46 | 38.00 | 38.00 |
| Ash | 1.20 | 1.63 | 1.08 | 1.13 | 1.44 | 1.61 | 2.08 |
| Protein (N×6.25) | 5.28 | 6.53 | 4.74 | 4.78 | 6.63 | 9.46 | 9.62 |
| Fat | 9.43 | 5.14 | 5.58 | 5.65 | 7.27 | 3.20 | 2.76 |
| Fiber | 0.27 | 0.22 | 0.30 | 0.44 | 0.30 | 0.22 | 1.93 |
| N-free extract | 60.20 | 64.48 | 63.26 | 66.28 | 59.90 | 47.51 | 45.61 |
| Starch | 21.01 | 20.30 | 16.23 | 11.94 | 15.00 | — | — |
| Invert sugar | 1.38 | 0.84 | 1.39 | 0.95 | 2.07 | — | — |
| Sucrose | 33.63 | 18.13 | 18.45 | 21.94 | 16.14 | — | — |
| Sodium chloride | 0.28 | 0.21 | 0.43 | 0.09 | 0.32 | — | — |
| Calcium | 0.04 | 0.04 | 0.03 | 0.04 | 0.04 | 0.08 | 0.04 |
| Phosphorus | 0.18 | 0.28 | 0.08 | 0.21 | 0.17 | 0.10 | 0.25 |
| Calories per pound | 1573 | 1498 | 1461 | 1520 | 1504 | 1164 | 1115 |

[1] Bak. Tech. 4, no. 10, p. 296, W. C. Luckow.
[2] Including icing.

Table III, giving the composition of several other products of which flour forms the basis, is inserted to show the relative food value of cake.

TABLE III

COMPOSITION OF VARIOUS FLOUR-CONTAINING PRODUCTS[1]

| | Water | Protein (N×6.25) | Fat | Fiber | Ash | Carbo-hydrates | Calories per pound |
|---|---|---|---|---|---|---|---|
| | % | % | % | % | % | % | |
| Crackers (water) | 6.4 | 11.7 | 5.0 | 0.4 | 1.2 | 75.3 | 1779 |
| " (soda) | 5.9 | 9.8 | 9.1 | 0.3 | 2.1 | 72.8 | 1874 |
| Cookies | 8.1 | 7.0 | 9.6 | 0.5 | 1.5 | 73.3 | 1857 |
| Doughnuts | 18.3 | 6.7 | 21.0 | 0.7 | 0.9 | 52.4 | 1941 |
| Macaroni | 12.0 | 13.0 | 1.4 | 0.4 | 0.6 | 72.6 | 1617 |
| " (cooked) | 78.4 | 3.0 | 1.5 | — | 1.3 | 15.8[3] | 402 |
| Noodles | 10.0 | 14.1 | 4.5 | 0.4 | 0.9 | 70.1 | 1708 |
| Gingerbread | 18.8 | 5.8 | 9.0 | 0.9 | 2.9 | 62.6 | 1624 |
| Zwieback | 5.8 | 9.8 | 9.9 | 0.8 | 1.0 | 72.7 | 1915 |
| Pilot bread | 8.7 | 11.1 | 5.0[2] | 0.3 | 1.0 | 73.9 | 1751 |
| Pretzels | 9.6 | 9.7 | 3.9 | 0.5 | 4.0 | 72.3 | 1657 |
| Pies (apple) | 42.5 | 3.1 | 9.8 | — | 1.8 | 42.8[3] | 1232 |
| " (squash) | 64.2 | 4.4 | 8.4 | — | 1.3 | 21.7[3] | 816 |
| Pie crust | 11.4 | 6.3 | 32.0 | 0.2 | 1.9 | 48.2 | 2298 |
| Matzoths | 6.0 | 8.5 | 1.0 | 0.2 | 0.4 | 83.9 | 1720 |
| Saltines | 5.6 | 10.6 | 12.7 | 0.5 | 2.6 | 68.0 | 1953 |
| Vanilla wafers | 6.7 | 6.6 | 14.0 | 0.3 | 1.1 | 71.3 | 1989 |
| Macaroons | 12.3 | 6.5 | 15.2 | 1.1 | 0.8 | 64.1 | 1920 |
| Ginger snaps | 6.3 | 6.5 | 8.6 | 0.7 | 2.6 | 75.3 | 1847 |
| Hot cross buns | 36.7 | 7.9 | 4.8 | — | 0.9 | 49.7[3] | 1241 |

[1] Chiefly from U. S. Department of Agriculture, Office of Experiment Stations Bulletin 28, Revised (1906), "The Chemical Composition of American Food Materials," p. 60–64, and Connecticut Agricultural Experiment Station Bulletin 286 (1927), "The Thirty-first Report on Food Products, etc.", p. 300–305.
[2] Pilot bread is often made without shortening.
[3] Includes fiber.

The materials used in cake making are all standard articles of food; flour is a good source of protein and an excellent source of energy; sugar and shortening are rich in energy values; eggs and milk are good sources of protein, minerals, fats, and vitamins; butter is rich in vitamins besides being an excellent source of energy.

In other words, protein is supplied by eggs, milk, and flour. Fat is furnished by eggs and shortening. Carbohydrates are present in abundance in flour and sugar. Vitamins are found in butter, milk, and eggs. Minerals are supplied by eggs and milk. Energy is furnished in large amounts by flour, sugar, and shortening, and to a less extent in eggs and milk. Since the constituents of cake contain minerals, fat, and protein in appreciable amounts and are rich in vitamins, cake may be considered good food, though as a rule it probably contains too large a proportion of carbohydrates or energy-giving constituents to form a prominent part of the diet.

## Cake Ingredients

The approximate percentage of ingredients used in making cake of the three different types is shown in Table IV.

TABLE IV

CAKE INGREDIENTS[1]

| | Angel food cake | Gold cake | Pound cake |
|---|---|---|---|
| | % | % | % |
| Flour | 15.6 | 23.9 | 24.8 |
| Egg whites | 41.3 | — | — |
| Egg yolk | — | 19.7 | — |
| Whole egg | — | — | 24.8 |
| Sugar | 41.8 | 27.0 | 24.8 |
| Leavening agent | 0.3[2] | 0.9 | 0.4 |
| Salt | 0.2 | 0.3 | 0.2 |
| Flavor | 0.3 | 2.1 | 0.1 |
| Shortening (water free) | — | 10.1[3] | 18.1[3] |
| Milk | — | 16.0 | 6.8 |

[1] These percentages may be varied considerably without injury to the quality of the cake. The figures given in the table represent an average formula which has given good results under laboratory conditions.

[2] Cream of tartar.

[3] If butter is used instead of vegetable shortening, increase the indicated weight by approximately one-fourth.

### *Flour*

Although flour constitutes less than one-fourth of the total ingredients used in cake, it is the basic constituent of all baked products. Each kind of flour may differ from other kinds not only in chemical composition but also in physical properties, such as granulation, water absorption capacity, gluten hydration capacity, and viscosity of flour-water suspensions. Each of these factors is believed to exert some influence on the character of the finished baked product. Table V shows the composition of different classes and grades of flour.

TABLE V

COMPOSITION OF VARIOUS COMMERCIAL GRADES OF HARD WHEAT FLOURS (YEARLY AVERAGES)[1]

| Year | Family Patent | | Short Patent | | Medium Patent | | Long Patent | | Straight Grade | | First Clear Fancy | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | Ash | Protein | Ash | Protein | Ash | Protein | Ash | Protein | Ash | Protein | Ash | Protein |
| | % | % | % | % | % | % | % | % | % | % | % | % |
| *Southwestern Flours* | | | | | | | | | | | | |
| 1927–28 | 0.370 | 10.20 | 0.390 | 10.60 | 0.410 | 10.80 | 0.430 | 11.05 | 0.450 | 11.28 | 0.580 | 11.71 |
| 1928–29 | .377 | 10.32 | .381 | 10.64 | .401 | 10.77 | .439 | 11.07 | .441 | 11.20 | .563 | 11.80 |
| 1929–30 | .360 | 10.54 | .387 | 10.92 | .407 | 11.09 | .426 | 11.32 | .455 | 11.40 | .591 | 12.02 |
| 1930–31 | | | .402 | 11.06 | .421 | 11.30 | .453 | 11.47 | .439 | 11.59 | .654 | 12.24 |
| 1931–32 | | | .390 | 10.73 | .410 | 11.03 | .433 | 11.22 | .453 | 11.34 | .651 | 12.05 |
| 1932–33 | | | .385 | 10.80 | .412 | 11.17 | .435 | 11.37 | .465 | 11.55 | .662 | 12.52 |
| 1933–34 | .380 | 10.50 | .395 | 11.46 | .420 | 11.67 | .440 | 11.89 | .432 | 12.10 | .645 | 13.21 |
| Grand Average 1927–1934 | .372 | 10.39 | .390 | 10.89 | .411 | 11.19 | .436 | 11.27 | .448 | 11.49 | .621 | 12.22 |
| *Northwestern Flours* | | | | | | | | | | | | |
| 1927–28 | | | 0.390 | 10.88 | 0.421 | 11.18 | 0.451 | 11.48 | 0.471 | 11.70 | 0.670 | 12.10 |
| 1928–29 | | | .390 | 10.75 | .420 | 11.05 | .450 | 11.35 | .470 | 11.65 | .670 | 12.15 |
| 1929–30 | | | .390 | 11.17 | .420 | 11.55 | .450 | 11.86 | .470 | 12.16 | .670 | 12.82 |
| 1930–31 | | | .400 | 11.67 | .420 | 12.00 | .450 | 12.25 | .470 | 12.55 | .684 | 13.33 |
| 1931–32 | | | .399 | 11.64 | .417 | 11.94 | .443 | 12.10 | .462 | 12.44 | .690 | 13.12 |
| 1932–33 | | | .396 | 11.85 | .420 | 12.10 | .440 | 12.30 | .470 | 12.70 | .695 | 13.55 |
| 1933–34 | | | .395 | 12.10 | .420 | 12.30 | .442 | 12.50 | .477 | 13.00 | .700 | 14.00 |
| Grand Average 1927–1934 | | | .394 | 11.44 | .419 | 11.80 | .449 | 11.98 | .470 | 12.31 | .683 | 13.01 |
| *Montana-Type Flours* | | | | | | | | | | | | |
| 1927–28 | | | 0.390 | 11.02 | 0.420 | 11.46 | 0.450 | 11.77 | 0.480 | 12.07 | | |
| 1928–29 | | | .390 | 11.36 | .420 | 11.59 | .450 | 11.82 | .480 | 12.12 | | |
| 1929–30 | | | .390 | 11.80 | .420 | 12.00 | .450 | 12.35 | .480 | 12.75 | | |
| 1930–31 | | | .390 | 12.53 | .420 | 12.80 | .450 | 13.06 | .480 | 13.46 | | |
| 1931–32 | | | .390 | 13.10 | .420 | 13.40 | .450 | 13.61 | .480 | 14.02 | | |
| 1932–33 | | | .417 | 12.85 | .420 | 13.25 | .450 | 13.60 | .480 | 14.05 | | |
| 1933–34 | | | .393 | 12.50 | .417 | 13.00 | .445 | 13.50 | .473 | 14.00 | | |
| Grand Average 1927–1934 | | | .394 | 12.16 | .419 | 12.34 | .449 | 12.81 | .479 | 13.21 | | |

[1] Private communication from Dr. E. E. Werner, St. Louis, Mo. These figures are averages of several thousand samples.

The composition and physical qualities of flour depend upon the character of the wheat used in milling. As is well known, wheat differs materially in composition, depending upon the class, the variety, the environment in which it is grown, and the conditions of the soil, fertilizer, etc. Therefore, as no two kinds of wheat have the same physical properties and chemical composition, they do not produce flours having identical composition and identical baking value. The baker is fully justified in seeking a flour having superior and uniform characteristics (both chemical and physical) for bread. He will likewise be rewarded by using the same care in selecting the flour best suited for cake.

A flour should be selected for the service which it is expected to render. This does not work a hardship upon the miller, for once the most suitable flour for any purpose has been found the miller can always produce a flour approaching it in quality. Knowing the quality of the various flours in regard to protein, ash, granulation, viscosity, pH, etc., the miller, by proper blending, can furnish practically any type of flour. The miller should know for what purposes each of his flours is most suitable.

Although knowledge of the chemical composition and physical properties of a flour is a great aid in forecasting its baking quality, the real test is the baking test. A flour that will make good bread probably will not be best suited for cake, but this can be proved only by a baking test.

Investigations conducted by the Bureau of Chemistry (Bur. Chem. Bull. 128, "Trilocal experiments on the influence of environment on the composition of wheat") showed that the composition of wheat varies with climatic conditions. In general the protein content of wheat during a year of high yield is appreciably lower than when, because of adverse climatic conditions, the yield of wheat is low. In other words, low yields and high protein content run parallel, and vice versa. It naturally follows, therefore, that the protein content of flour varies.

### *Shortening*

The shortenings ordinarily used for cake are butter, lard, and the various hydrogenated vegetable products. Butter is generally considered the ideal shortening for cake, especially because of the pleasing flavor it imparts to the product. Butter contains about 16% water and at least 80% fat, hence more of it must be used than of lard or vegetable shortening, which are 100% fat. Shortening makes cake tender and improves the grain, texture, and keeping quality. A cake is said to vary in richness in accordance with the amount of shortening used, *e.g.*, the old-fashioned pound cake is considered the richest because it

has the largest amount of shortening, while angel food is the other extreme. The cost of the shortening is about 20% of the total cost of the ingredients in cake. Generally speaking, the keeping quality of cakes varies directly with the shortening content.

### *Eggs*

The item of greatest expense in cake making is eggs, either whole eggs, whites, or yolks—fresh, frozen, or dried. The cost of the eggs constitutes approximately one-half the total cost of the ingredients. That is one reason why there is such a temptation to curtail the amount of eggs even at the risk of producing an inferior cake. Besides imparting richness and improving the grain, texture, flavor, and general appearance of the finished product, eggs serve to hold the air and build the structure of the cake. They also increase the lightness of the cake and give it a moisture-retaining property. They aid in delaying crumbling, which is one indication of staling. Containing a high amount of fat, the yolk has also considerable shortening value. Being rich in protein, fat, minerals, and vitamins, eggs supply more real food value to cake than does any other one constituent.

Eggs are made up of approximately 10.5% shell, 58% white, and 31.5% yolk.

Table VI shows the composition of the edible portion of eggs.

TABLE VI

COMPOSITION OF EGGS—EDIBLE PORTION[1]

| Substance | Water | Protein | Fat | Ash |
|---|---|---|---|---|
| | % | % | % | % |
| Whole egg | 73.7 | 14.8 | 10.5 | 1.0 |
| Egg white | 86.2 | 13.0 | 0.2 | 0.6 |
| Egg yolk | 49.5 | 16.1 | 33.3 | 1.1 |

[1] U. S. Department Agriculture, Office of Experiment Stations Bulletin 28, revised 1906.

Eggs may be used in cake making so long as they are in an edible condition. Strictly fresh eggs are not essential; good cold storage eggs are quite satisfactory, although it is not always possible to obtain storage eggs of uniform quality.

Frozen egg whites, yolks, and mixed whites and yolks are frequently used by commercial bakers. The advantage of using frozen eggs is that a product of uniform quality is available throughout the year.

In freezing either whole eggs or egg yolks, the eggs are broken, 5 to 8% glycerine[2] or cane sugar to the extent of 10% is added, and

[2] Glycerine is not a recognized food and is not recommended.

the mixture is thoroughly churned. It is then placed in tin cans and frozen solid, after which it is hermetically sealed and kept at a temperature of about 15° F. until used. As some of the white adheres to the shell, about 3 to 4% of the egg is lost in the breaking process.

The composition of frozen whole eggs, frozen yolks, and frozen whites is essentially the same as that of the corresponding fresh product except for the sugar or glycerine added before freezing. The addition of these substances is said to improve the consistence of the thawed product, making it more nearly like the fresh product. Some bakers claim that glycerine not only improves the quality of the cake but also aids in the retention of moisture.

Although the quality of the frozen eggs is more uniform than that of fresh eggs, bakers have found that a somewhat better cake is obtained with first-class, fresh, shell-eggs, and that both fresh eggs and frozen eggs are far superior to dried eggs for baking purposes. These results have been corroborated in this laboratory.

The composition of dried egg products, according to analyses made by the Bureau of Chemistry, is shown in Table VII.

TABLE VII

ANALYSES OF DRIED EGG PRODUCTS[1]

| Product | Moisture | Protein N×6.25 | Alcohol precipitable nitrogen | Total nitrogen | Lipoids | Lipoid $P_2O_5$ |
|---|---|---|---|---|---|---|
| | % | % | % | % | % | % |
| Whole egg (8 samples) | | | | | | |
| Maximum | 9.30 | 46.75 | 3.32 | 7.48 | 48.79 | 1.35 |
| Minimum | 5.48 | 43.75 | 2.81 | 6.98 | 42.03 | 1.16 |
| Average | 7.05 | 45.52 | 3.04 | 7.22 | 46.09 | 1.25 |
| Whites (6 samples) | | | | | | |
| Maximum | 16.15 | 76.94 | 9.48 | 12.31 | — | — |
| Minimum | 11.65 | 74.62 | 8.69 | 11.94 | — | — |
| Average | 14.37 | 75.91 | 8.99 | 12.14 | — | — |
| Yolks (5 samples) | | | | | | |
| Maximum | 4.68 | 33.44 | 0.84 | 5.35 | 64.03 | 1.72 |
| Minimum | 2.93 | 32.56 | 0.54 | 5.13 | 62.75 | 1.66 |
| Average | 4.15 | 32.81 | 0.71 | 5.25 | 63.36 | 1.67 |

[1] Unpublished results—Bureau of Chemistry.

In drying eggs, about 90% of the water is removed. Approximately 3½ pounds of liquid whole eggs, 7¼ pounds of liquid whites, and 2¼ pounds of liquid yolks are required to yield 1 pound of the respective dried products.

Dried eggs are reconstituted to simulate fresh eggs by adding water. One part of dried whole egg and 2½ parts water will yield a product having essentially the same composition as fresh whole eggs. In practice, it is advantageous to use 3 parts of water.

To reconstitute dried yolk the ratio should be 1 part yolk and 1 part water, though here again it is more practicable to use 1½ parts water. In the same way, mixing 1 part dried egg albumen with 6 to 7 parts water will produce a product very similar in composition to fresh whites. All reconstituted egg products should be used as soon as possible as they are very perishable.

*Sugar*

Sugar is the product used in greatest quantity or proportion in cake making; some cakes contain as much as 40% sugar, and few contain less than 18%. The amount of sugar, except in pound cake, is always in excess of the shortening. In the cake industry as a whole, 35% more sugar than flour is used and the value of the sugar is nearly twice that of the flour. Sugar is therefore one of the most important constituents of cake.

Too much sugar in cake may cause a sugary and thick crust. On the other hand a mixture containing too little sugar and shortening in proportion to the flour may produce a cake having large holes and a texture simulating bread.

The physical properties of sugar play an important role in cake making.

The use of a powdered sugar, a fine granulated, or a coarse granulated sugar may make an appreciable difference in the quality of the final product. Fine granulated sugar is generally preferred for cake making.

Besides sucrose (cane and beet sugar), which is the most common form of sugar, dextrose (corn sugar), and maltose (malt sugar) may be used to make good cake. Corn sugar is said to be approximately two-thirds as sweet as cane sugar or about as sweet as malt sugar. These sugars of low sweetening power are often used in fruit and spice cakes, which need not be especially sweet. Besides increasing the keeping quality, malt sugar and honey impart certain desirable flavors to cake. Honey is about 75% invert sugar, which is composed of dextrose and levulose. Levulose itself is considerably sweeter than cane sugar. Molasses, which is a low grade form of sugar, is used to impart color as well as sweetness to cake. It also gives moisture-retaining qualities. Molasses contains about 20% water, 35% crystallizable sugar, 35% invert sugar, 5% organic or extractive matter, and 5% ash.

Invert sugar can be prepared by boiling at 234° F. 20 pounds of cane or beet sugar in 7 pounds of water containing one-half ounce tartaric acid. As stated, invert sugar improves flavor and helps the cake to retain moisture and remain fresh for a considerable period.

Replacing 3 to 10% of the cane or beet sugar with invert sugar is enough to give cake moisture-retaining properties.

### *Milk*

Milk in any of its various forms—liquid, dry, evaporated, or condensed—may be used in making practically all kinds of cake except angel food cake. While one of the minor constituents, and not absolutely necessary, as it can often be replaced by water, milk constitutes more than 3% of the total cost of the ingredients used in making cake. It is used chiefly to replace eggs. Although not a normal constituent of certain cakes, as for example pound cake, it is often used therein, especially when butter, which contains at least 16% water, is replaced by a vegetable shortening, which is water-free. Whether liquid milk or one of its modifications is used is chiefly a matter of availability and price. In using dry milk, it is always better to reconstitute it than to attempt to incorporate the dry powder into the cake mixture. When liquid milk is used, care should be taken that it is not too cold, as in that case it will congeal the shortening thus causing the finished cake to have coarse uneven grain.

The composition of milk products is given in Table VIII.

TABLE VIII

COMPOSITION OF MILK PRODUCTS[1]

| Kind of product | Water | Protein N×6.38 | Fat | Ash | Lactose | Sucrose |
|---|---|---|---|---|---|---|
| | % | % | % | % | % | % |
| Liquid whole milk | 87.1 | 3.2 | 3.9 | 0.7 | 5.1 | |
| Liquid skim milk | 90.3 | 3.6 | 0.1 | 0.8 | 5.2 | |
| Evaporated whole milk | 73.0 | 6.7 | 8.2 | 1.4 | 10.7 | |
| Evaporated skim milk | 71.0 | 10.8 | 0.3 | 2.4 | 15.5 | |
| Sweet condensed whole milk | 28.0 | 7.5 | 9.2 | 1.7 | 12.0 | 41.6 |
| Sweet condensed skim milk | 29.0 | 9.2 | 0.3 | 2.0 | 13.4 | 46.1 |
| Dry whole milk[2] | 3.0 | 24.1 | 29.3 | 5.3 | 38.3 | |
| Dry skim milk[3] | 3.0 | 36.0 | 1.0 | 8.0 | 52.0 | |

[1] Typical analyses compiled from various sources.

[2] Theoretically one part of dry whole milk when mixed with 7 parts of water will form a product similar in composition to liquid whole milk. To reconstitute milk, 1 pound of powdered whole milk is usually added to 7½ pints of water. In other words, for every quart of water, use 4¼ ounces of dry whole milk powder.

[3] To reconstitute skim milk, 12 ounces of skim milk powder should be added to about 7¾ pints of water. One pound of sweetened condensed skim milk is equivalent to a mixture of 4½ ounces powdered skim milk, 7 ounces of sugar, and 4½ ounces of water.

### *Leavening Materials*

It has been estimated that the baking powder and cream of tartar used in making commercial cake constitute together about 1.5% of the value of all the ingredients therein employed. Cream of tartar is the usual acid substance employed in making angel food cake, although other weak acid-reacting substances are sometimes used.

Baking powders may be classed as—slow acting, fast acting, double acting. The first type may contain sodium acid pyrophosphate or sodium aluminum sulfate; the acid constituents of the second are calcium acid phosphate or potassium acid tartrate with or without tartaric acid; the third is composed of a combination of slow and fast acting ingredients. Sour milk or buttermilk and even molasses are occasionally used as acid constituents in making cake, as all these substances contain acids which will react with soda and thus produce a portion of the leavening. Accordingly, less baking powder is required when such products are employed. When 20 pounds (not quite 2½ gallons) of buttermilk or sour milk of the proper degree of acidity are combined with 4 ounces of soda, as much carbon dioxide gas will be produced as will be produced from 1 pound of baking powder. If a somewhat more alkaline or less acid product is desired, for example, when making devil's food cake, the amount of buttermilk may be decreased to 14 pounds instead of 20 pounds. When cream of tartar and soda are used, approximately 2 parts of the former should be mixed with 1 of the latter. Of course, all leavening agents should be thoroughly incorporated with the mixture, otherwise large and uneven holes will result. Too much leavening agent has a tendency to hasten the staling of cake. To retard this process of deterioration some bakers use small amounts of glucose, honey, glycerine, etc.

### *Flavoring Materials*

While most flavoring materials possess little or no food value they may rightly be considered among the essential constituents of cake. Flavoring materials constitute more than 5% of the cost of all materials used in commercial cake making. The principal flavoring materials used are the various spices, such as mace, cinnamon, ginger, and the extracts of almond, vanilla, orange, and lemon. Orange or lemon oil may be used instead of the corresponding extract. Dried orange peel can also be used with satisfaction.

Flavors are used for a double purpose. They not only give a pleasing and satisfactory effect, enjoyed by nearly everybody, but they also conceal the taste of certain necessary and/or desirable constituents, such as the newly developed "fish flour," which is desirable because it is rich in calcium and phosphorus. When fish flour is used in cookies even up to 10 to 15% of the weight of the flour its taste can be entirely concealed by using sufficient orange oil as a flavor. It should be remembered, however, that many an otherwise good cake is injured or ruined by too much flavor.

The following are some of the definitions and standards for spices and flavoring materials for food purposes given in Service and Regu-

latory Announcements of the Food and Drug Administration, U. S. Department of Agriculture:[3]

"Cinnamon is the dried bark of cultivated varieties of Cinnamomum zeylanicum Nees or of C. cassia (L) Blume, from which the outer layers may or may not have been removed."

"Ground cinnamon, ground cassia, is the powder made from cinnamon. It contains not more than 5 percent of total ash, nor more than 2 percent of ash insoluble in hydrochloric acid."

"Ginger is the washed and dried, or decorticated and dried, rhizome of *Zingiber officinale* Roscoe. It contains not less than 42 percent of starch, not more than 8 percent of crude fiber, not more than 1 percent of lime (CaO), not less than 12 percent of cold water extract, not more than 7 percent of total ash, not more than 2 percent of ash insoluble in hydrochloric acid nor less than 2 percent of ash soluble in cold water."

"Mace is the dried arillus of *Myristica fragrans* Houtt. It contains not less than 20 percent nor more than 30 percent of non-volatile ether extract, not more than 10 percent of crude fiber, not more than 3 percent of total ash, nor more than 0.5 percent of ash insoluble in hydrochloric acid."

"Lemon extract is the flavoring extract prepared from oil of lemon, or from lemon peel, or both and contains not less than 5 percent by volume of oil of lemon."

"Oil of lemon is the volatile oil expressed, without the aid of heat, from the fresh peel of the lemon (*Citrus limonia* Osbeck), with or without previous separation of the pulp and peel."

"Orange extract is the flavoring extract prepared from oil of orange, or from orange peel, or both, and contains not less than 5 percent by volume of oil of orange."

"Oil of orange is the volatile oil obtained by expression or alcoholic solution from the fresh peel of the orange (*Citrus aurantium* L.) and has an optical rotation (25° C.) of not less than +95° in a 100 mm. tube."

"Vanilla extract is the flavoring extract prepared from vanilla bean, with or without one or more of the following: sugar, dextrose, glycerine. It contains in 100 cc. the soluble matters from not less than 10 grams of the vanilla bean."

"Vanilla bean is the dried, cured fruit of *Vanilla fragrans* (Salish) Ames (V. planifolia Andr.)."

"Almond extract is the flavoring extract prepared from oil of bitter almonds, free from hydrocyanic acid, and contains not less than 1 percent by volume of oil of bitter almonds."

"Oil of bitter almonds, commercial, is the volatile oil obtained from the seed of the bitter almond (*Amygdalus communis* L.), the apricot (*Prunus armeniaca* L.) or the peach (*Amygdalus persica* L.)."

"Allspice, pimento, is the dried, nearly ripe fruit of *Pimenta officinalis* Lindl. It contains not less than 8 percent of quercitannic acid (calculated from the total oxygen absorbed by the aqueous extract), not more than 0.4 percent of ash insoluble in hydrochloric acid."

"Cloves are the dried flower buds of *Caryophyllus aromaticus* L. They contain not more than 5 percent of cloves stems, not less than 15 percent of volatile ether extract, not less than 12 percent of quercitannic acid (calculated from the total oxygen absorbed by the aqueous extract), not more than 10 percent of crude fiber, not more than 7 percent of total ash, nor more than 0.5 percent of ash insoluble in hydrochloric acid."

"Nutmeg is the dried seed of *Myristica fragrans* Houtt, deprived of its testa, with or without a thin coating of lime (CaO). It contains not less than 25 percent of non-volatile ether extract, nor more than 10 percent of crude fiber, not more than 5 percent of total ash, nor more than 0.5 percent of ash insoluble in hydrochloric acid."

### *Salt*

Salt is used in very small amounts, but it is nevertheless essential for a well-flavored cake. Wherever a vegetable shortening is used instead of butter, the amount of salt should be increased somewhat, as butter normally contains 1½ to 2% of salt and the vegetable shortening

[3] S. R. A., F. D. No. 2, Rev. 4, Aug. 1933.

contains none. Too much salt, however, has an adverse effect upon the size of the cake as well as on the flavor.

### Starch and Other Ingredients

Starch is often used with certain strong flours to reduce the effect of the strong gluten and to make these flours in a sense comparable to soft wheat flour. The starch ordinarily used for this purpose in this country is that made from corn because it is the most readily available and the cheapest; but any starch and in fact any other non-glutenous food material, such as corn flour, soybean flour, wheat germ, artichoke flour, potato flour, all of which differ considerably in composition and physical characteristics from wheat flour and from each other, can be added to a strong flour to make it more suitable for cake making. Even whole wheat flour may be used for this purpose.

The market price, both wholesale and retail, of the various ingredients used in cake making is given in Table IX.

TABLE IX

WHOLESALE AND RETAIL PRICES OF CAKE INGREDIENTS[1]

| Substance | Wholesale | Retail |
|---|---|---|
| | *Cents per pound* | *Cents per pound* |
| Flour | 2.55 | 3.0 |
| Egg white | 14.0 | 15.4 |
| Egg yolk | 18.0 | 19.8 |
| Whole egg | 15.0 | 16.5 |
| Sugar | 4.2 | 5.0 |
| Vegetable shortening hydrogenated | 13.5 | 23.0 |
| Butter | 26.0 | 27.0 |
| Milk, fluid | 10.0[2] | 14.0[2] |
| Milk, dry | — | 30.0 |
| Cream of tartar | 40.0 | 72.0 |
| Baking powder | 17.0 | 32.0 |
| Vanilla | 125.0[3] | 320.0[3] |
| Lemon juice | 24.0[2] | 53.0[2] |
| Mace | 60.0 | 184.0 |
| Salt | 0.85 | 2.22 |

[1] As of March 1932.
[2] Cents per quart.
[3] Cents per pint.

### Cost of Ingredients

With the wholesale price of ingredients as given in Table IX, the cost of materials entering into commercial cake is approximately $100,000,000 annually. The cost of flour, the basic ingredient, constitutes less than 7% or one-fourteenth of this total; the cost of eggs nearly one-half (48%); shortening nearly one-fifth (18.8%); sugar more than one-seventh (15.4%), and milk more than one-twentieth (5.5%).

The quantity and cost of ingredients utilized annually in commercial cake making are shown in Table X.

TABLE X

QUANTITY AND COST OF COMMERCIAL CAKE INGREDIENTS

| Ingredients | Amount (000 omitted) | Wholesale[1] price per unit | Value (000 omitted) | Percent of total |
|---|---|---|---|---|
| Flour, *bbls.* | 1,325 | $5.00 | $6,625 | 6.8 |
| Whole eggs (fresh), *lbs.* | 97,000 | .12 | 11,640 | 11.8 |
| Egg whites (frozen), *lbs.* | 149,500 | .14 | 20,930 | 21.2 |
| Egg yolk (frozen), *lbs.* | 83,700 | .18 | 15,066 | 15.3 |
| Sugar, *lbs.* | 360,500 | .042 | 15,141 | 15.4 |
| Shortening (veg.), *lbs.* | 75,500 | .095 | 7,172 | 7.2 |
| Butter, *lbs.* | 44,300 | .26 | 11,518 | 11.6 |
| Milk, *lbs.* | 108,800 | .05 | 5,440 | 5.5 |
| Baking powder, *lbs.* | 4,900 | .17 | 836 | 0.9 |
| Cream of tartar, *lbs.* | 1,100 | .40 | 440 | 0.4 |
| Salt, *lbs.* | 2,300 | .0085 | 19 | — |
| Concentrated flavoring, *lbs.* | 2,250 | 1.25 | 2,750 | 2.8 |
| Lemon juice, *lbs.* | 9,000 | .12 | 1,080 | 1.1 |
| Mace and other spices, *lbs.* | 80 | .60 | 48 | — |

[1] As of March, 1932.

TABLE XI

INGREDIENTS AND COST PER POUND OF CAKE[1]

| Ingredients | Weight | Cost | | | |
|---|---|---|---|---|---|
| | | Wholesale | | Retail | |
| | *Grams* | *Cents* | *%* | *Cents* | *%* |
| | | *Angel food cake* | | | |
| Flour | 82.8 | .456 | 4.59 | .547 | 4.61 |
| Egg white | 221.6 | 6.840 | 68.89 | 7.521 | 63.34 |
| Sugar | 221.6 | 2.052 | 20.66 | 2.442 | 20.57 |
| Cream of tartar | 1.9 | .168 | 1.69 | .302 | 2.54 |
| Salt | 1.0 | .002 | .02 | .005 | .04 |
| Vanilla | 1.5 | .412 | 4.15 | 1.057 | 8.90 |
| Total | 530.4 | 9.930 | 100.00 | 11.874 | 100.00 |
| | | *Gold cake* | | | |
| Flour | 122.6 | .689 | 7.76 | .811 | 7.01 |
| Egg yolk | 101.6 | 4.032 | 45.42 | 4.435 | 38.32 |
| Sugar | 139.1 | 1.288 | 14.51 | 1.533 | 13.24 |
| Vegetable shortening (hydrogenated) | 52.2 | 1.553 | 17.50 | 2.648 | 22.88 |
| Milk | 82.4 | .871 | 9.81 | 1.220 | 10.54 |
| Leavening | 4.4 | .165 | 1.86 | .310 | 2.68 |
| Flavor (lemon juice) | 10.9 | .276 | 3.11 | .610 | 5.27 |
| Salt | 1.4 | .003 | .03 | .007 | .06 |
| Total | 514.6 | 8.877 | 100.00 | 11.574 | 100.00 |
| | | *Pound cake* | | | |
| Flour | 125.8 | .708 | 7.66 | .833 | 6.81 |
| Eggs | 125.8 | 4.160 | 45.04 | 4.577 | 37.43 |
| Sugar | 125.8 | 1.165 | 12.61 | 1.387 | 11.34 |
| Vegetable shortening (hydrogenated) | 91.6 | 2.727 | 29.52 | 4.644 | 37.98 |
| Milk | 34.7 | .367 | 3.97 | .513 | 4.20 |
| Baking powder | 1.5 | .056 | .61 | .108 | .88 |
| Mace | .4 | .053 | .57 | .162 | 1.32 |
| Salt | 1.1 | .002 | .02 | .005 | .04 |
| Total | 506.7 | 9.238 | 100.00 | 12.229 | 100.00 |

[1] As of March 1932.

At wholesale prices, the cost of ingredients used in making 1 pound of cake of the 3 types studied ranges from approximately 8.9 cents to 9.9 cents, gold cake being the cheapest and angel food the most expensive. At retail prices, the cost of ingredients is 2 to 3 cents more per pound of cake than at wholesale prices. The retail prices given in Table XI prevailed in Chicago at approximately the same time that the wholesale prices were obtained. The amounts of the various ingredients are estimates based upon our laboratory formulas.

Since the cost of the flour is only a trifle when compared to the total cost of the ingredients in cake, one should use only such flours as are best adapted for the type of cake desired. The most expensive ingredient is eggs. This ingredient makes up approximately 65% of the cost of the materials used in angel food, and 40% in gold and pound cake.

## Formulas

Cake of good quality can be made in innumerable ways. The formulas given in Tables XII and XIII are arranged according to the amount of shortening used, pound cake having the most and sponge cake and angel food cake having none.

TABLE XII

CAKE FORMULAS[1]

| Kind of cake | Proportions of ingredients are based on flour as 100 | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| | Flour | Vegetable shortening | Sugar | Eggs | Milk | Baking powder | Salt | Starch | Total of shortening, sugar, milk, and eggs |
| Old-fashioned pound | 100 | 80[2] | 100 | 100 | 15 | — | 2.5 | — | 295 |
| Laboratory pound | 100 | 73 | 100 | 100 | 45 | 1.4 | 0.6 | — | 318 |
| Commercial pound | 100 | 50 | 100 | 50 | 50 | — | 1.5 | — | 250 |
| White pound | 100 | 50 | 3+96[3] | 50[4] | 50 | — | 1.6 | — | 249 |
| Package | 100 | 45 | 95 | 45 | 50 | 1.5 | 1.8 | — | 235 |
| Laboratory gold | 100 | 42 | 113 | 83[5] | 67 | 3.6 | 0.9 | — | 305 |
| Cup | 100 | 40 | 85 | 30 | 60 | 3.0 | 2.0 | — | 215 |
| White | 100 | 33 | 78 | 2[6] | 7[7] | — | 1.3 | — | 120 |
| Layer | 100 | 31 | 92 | 30[4] | 70 | 3.5 | 1.5 | — | 223 |
| Short sponge | 100 | 20 | 120 | 90[4] | 45 | 1.5 | 1.8 | — | 275 |
| Sponge | 100 | — | 130 | 200[4] | — | — | 1.6 | — | 330 |
| Laboratory angel food | 100 | — | 231[8] | 231[4] | — | 1.9[9] | 1.4 | — | 462 |
| Commercial angel food | 100 | — | 229[10] | 224[4] | — | 3.5[9] | 1.7 | 14 | 453 |

[1] Flavoring of suitable kind and quantity is added to these cakes, but not included in these formulas.
[2] If butter is used in place of vegetable shortening, increase the indicated weight by approximately one-fourth, and decrease the milk to allow for the water contained in the butter.
[3] Use 3 parts of invert sugar and 96 parts of granulated sugar.
[4] Egg whites.
[5] Egg yolks.
[6] Albumen (dry).
[7] Powdered milk.
[8] One-fourth powdered and three-fourths granulated sugar.
[9] Cream of tartar.
[10] Powdered sugar.

TABLE XIII

INGREDIENTS AND PERCENTAGE COMPOSITION OF CAKES

| Ingredients | Quantity | | Composition | | | | |
|---|---|---|---|---|---|---|---|
| | | | Water | Protein | Fat | Carbohydrates | Ash |
| | *Grams* | % | % | % | % | % | % |
| | | | *Angel food cake* | | | | |
| Flour | 82.5 | 15.6 | 13.8 | 8.0 | 1.4 | 76.4 | 0.4 |
| Egg white | 221.6 | 41.8 | 86.2 | 13.0 | 0.2 | — | 0.6 |
| Sugar | 221.6 | 41.8 | — | — | — | 100.0 | — |
| Cream of tartar | 1.9 | 0.3 | 63.0[1] | — | — | — | 37.0 |
| Vanilla | 1.5 | 0.3 | 80.0[2] | — | — | 19.0[3] | 0.2 |
| Salt | 1.0 | 0.2 | — | — | — | — | 100.0 |
| | | | *Gold cake* | | | | |
| Flour | 122.6 | 23.9 | 13.8 | 9.0 | 1.4 | 75.4 | 0.4 |
| Egg yolk | 101.6 | 19.7 | 49.5 | 16.1 | 33.3 | — | 1.1 |
| Sugar | 139.1 | 27.0 | — | — | — | 100.0 | — |
| Vegetable shortening | 52.2 | 10.1 | — | — | 100.0 | — | — |
| Milk | 82.4 | 16.0 | 87.1 | 3.2 | 3.9 | 5.1 | 0.7 |
| Baking powder | 4.4 | 0.9 | 14.0[4] | — | — | 40.0 | 46.0 |
| Lemon juice | 10.9 | 2.1 | 91.0 | — | — | 8.5[5] | 0.5 |
| Salt | 1.4 | 0.3 | — | — | — | — | 100.0 |
| | | | *Pound cake* | | | | |
| Flour | 125.8 | 24.8 | 13.8 | 10.0 | 1.4 | 74.3 | 0.5 |
| Whole egg | 125.8 | 24.8 | 73.7 | 14.8 | 10.5 | — | 1.0 |
| Sugar | 125.8 | 24.8 | — | — | — | 100.0 | — |
| Vegetable shortening | 91.6 | 18.1 | — | — | 100.0 | — | — |
| Milk | 34.7 | 6.8 | 87.1 | 3.2 | 3.9 | 5.1 | 0.7 |
| Baking powder | 1.5 | 0.4 | 14.0[4] | — | — | 40.0 | 46.0 |
| Mace | 0.4 | 0.1 | — | — | — | — | — |
| Salt | 1.1 | 0.2 | — | — | — | — | 100.0 |

[1] Volatile = $CO_2 + H_2O$.
[2] Volatile, including 50–60% alcohol.
[3] Includes extractive matter.
[4] Volatile = $CO_2$.
[5] Includes 6 to 7% citric acid.

To a certain extent, the formulas given in Tables XII and XIII may be considered average, that is, they will make very satisfactory cake, and yet it should be remembered that each of these formulas can be changed appreciably in respect to the quantity of every one of the ingredients, making it possible for one cake to have certain characteristics different from every other cake of the same type.

Table XIV gives the average composition of batter and of cake of the three types studied.

The foregoing formulas indicate that, in general, the housewife is somewhat more lavish in the use of the expensive ingredients than is the commercial baker. Old-fashioned pound cake furnishes an example. In this case, the housewife generally uses equal parts of sugar, flour, eggs, and butter, while the commercial baker's formulas call for appreciably less butter and eggs and the inclusion of vegetable shortening and milk. This difference in the formula makes a difference in the taste and quality of the cake, as the eggs and butter both add flavor as well as improve the texture and general appearance.

TABLE XIV

COMPOSITION OF BATTER AND CAKE

| Type of cake | Weight | Water | Protein | Fat | Carbohydrates | Ash | Calories per pound |
|---|---|---|---|---|---|---|---|
| | *Grams* | % | % | % | % | % | |
| | | | *Batter* | | | | |
| Angel food | 530.4 | 38.6 | 6.7 | 0.3 | 53.7 | 0.6 | — |
| Gold | 514.6 | 29.1 | 5.9 | 17.7 | 46.3 | 1.0 | — |
| Pound | 506.7 | 27.7 | 6.4 | 21.3 | 43.7 | 0.8 | — |
| | | | *Cake* | | | | |
| Angel food | 453.6 | 28.2[4] | 7.8 | 0.4 | 62.8[1] | 0.7 | 1006 |
| Gold | 453.6 | 19.5 | 6.6 | 20.1 | 52.5[2] | 1.2 | 899 |
| Pound | 453.6 | 19.3 | 7.1 | 23.8 | 48.9[3] | 0.9 | 929 |

[1] Includes 48.9% cane sugar.
[2] Includes 30.6% cane sugar.
[3] Includes 27.7% cane sugar.
[4] Loss in weight during baking: angel food cake, 76.9 g.; gold cake, 61 g.; pound cake 53.1 g.; or 14.5, 11.9, and 10.5%, respectively.

The amount of shortening, eggs, milk, and sugar varies greatly in the different kinds of cake. Angel food cake, for example, carries about 4½ times as much egg and sugar as flour; pound cake comes next with 2.5 to 2.9 times as much of these ingredients as of flour. White cake is the "leanest" cake in the group as it contains only 1.2 times as much egg and sugar as flour.

Such a large amount of non-flour materials can be used in making angel food cake because of the leavening power of the egg white. In pound cake the flour really helps carry the heavy burden of sugar, shortening, and eggs and that is why the flour used for pound cake should be moderately strong. It should again be noted that while the old-fashioned pound cake employs 4.8 times as much of the combined eggs, shortening, and sugar as of flour, the ordinary commercial pound cake carries only 2.5 times as much of these ingredients. In certain cakes milk can be substituted in part for eggs, 1 part of the former replacing 1 of the latter. When the flour used is somewhat too strong, it is advisable to increase the amount of sugar and shortening in the proportion of 2 parts of sugar to 1 part of shortening or to add starch, soybean flour, or other non-glutinous flour.

## Cake Making Procedure

Cake making is even more empirical than bread making and neither can be placed in the category with chemical analysis. The varying results obtained in cake making may possibly be due to the fact that the same exactness of manipulation cannot be given to baking as is given to chemical analysis. By utilizing the same materials and operating as nearly as possible in the same manner throughout the process of cake making, one would expect to obtain duplicate results.

But, when the flours used have been milled from different wheats and in different mills, when the eggs have been produced by hens fed with different feeds, and when the other materials are produced under varying conditions and consequently are not the same in composition, the duplication of results becomes difficult if not impossible.

In addition to the variations in the compositions of the ingredients used, there are variations in the methods of mixing, the temperature of the mixture, the relative proportions of the ingredients, the temperature of the oven, and the time of baking at the different temperatures. Each has its effect.

### *Mixing*

Different types of cake require different methods of mixing. The foam type depends for its lightness upon the incorporation of air in the mixed mass, which expands during the baking process. Consequently with this type of cake every precaution should be taken to insure the formation of an abundance of small air bubbles and to avoid any condition that will cause the loss of this entrapped air until the frame structure of the cake has become set.

Thus, in the laboratory method of making angel food cake, the egg white to which salt has been added is whipped to a froth, the cream of tartar is added, and the whipping is continued until a moderately stiff foam is formed. The fine granulated sugar is then gradually added while the beating is continued at low speed. When the foam is stiff enough to hold its shape and before it becomes dry, the beating is stopped and the flour and powdered sugar are very carefully folded in, so as to cause as little loss as possible of the entrapped air. Next the batter is cautiously transferred to a suitable pan which has just been moistened with cold water, and the cake is baked immediately.

The time required to beat-up the egg white depends upon its temperature, the consistency, and pH. If the egg white is thick, its whipping quality will be improved by diluting with 10 to 12% of water. The addition of cream of tartar or other acid reacting substance to reduce the pH to 6.5 or less will greatly shorten the time required to produce a foam suitable for making angel food cake. Ordinarily the egg white can be whipped with a mechanical beater in 3 to 5 minutes if proper conditions are maintained. The whipping should be continued until the egg white will just stand up in points when the beater is removed, but it should not be allowed to become dry and fluffy. If the whipping is carried too far the finished product will be of small volume and the grain, texture, and color will be somewhat impaired.

### *Creaming Method*

With the heavier types of cakes the method of mixing may be somewhat varied and still produce satisfactory results. Much has been written about the thorough "creaming" of the fat and sugar and the proper order in which the different ingredients should be added. Frequently directions are given to "cream" the sugar and shortening to the lightest possible consistency, then gradually incorporate the eggs into this beaten mass or whip the eggs separately and add them to the creamed shortening and sugar, after which add gradually and alternately the milk and flour. During the mixing process the water in the eggs and milk produces an emulsion with the shortening. If the eggs and milk are added gradually, the shortening forms the outer phase, and a water-in-fat dispersion results. In baking the cake some of this moisture is vaporized and in a large measure furnishes the leavening of the cake.

If the eggs and milk are added too rapidly, the phase of the emulsion may change from a water-in-fat to a fat-in-water dispersion, and there results a curdled mass that presents anything but an attractive appearance. The addition of the flour to this curdled mass, however, brings about a smooth mixture, and the quality of the cake baked therefrom is practically as good as that made from a non-curdled batter.

### *"Single Stage" Method*

We have demonstrated in the course of this work that excellent cakes of the heavier types can be made by what is known as the "single stage" process of mixing, in which all the ingredients with the exception of the leavening are added at once. In case cream of tartar and soda are used instead of baking powder the soda may be added with the flour and the cream of tartar reserved until near the end of the mixing. When all the ingredients are in the mixing bowl, the mixer is started at low speed and continued until the flour is no longer dusty (½ min.), then the speed is changed from low to medium and the mixing is continued for 10 minutes. At this stage the baking powder or cream of tartar, as the case may be, is added, and the mixing continued for 1 to 2 minutes longer. The leavening should be uniformly distributed, but mixing should not be continued any longer than necessary to accomplish this purpose, as some of the gas from the leavening will escape. The proper temperature should be maintained while the mixing is in progress. Our experiments indicate that it is preferable to mix the alkaline ingredient, *viz.*, the soda, with the flour, egg, milk, etc., as a better emulsion is formed if the mixture is slightly alkaline. The acid ingredient is not added until 1 to 2 minutes before the mixing is complete.

This "single stage" method of mixing saves time and is subject to fewer variations than the "creaming" method. Its use makes for uniformity of production.

A similar method of mixing was suggested by Denton and Miller,[4] but no mention was made of the desirability of withholding the baking powder or one of the leavening constituents from the mixture until near the end of the process. This is an important factor in this type of cake making, as otherwise the loss of carbon dioxide may be so great that the cake will not be properly leavened.

### *Speed of Mixing*

In making angel food cake the speed of mixing affects the character of the finished product. Ordinarily, high speed is used in beating up egg white, low speed is employed in mixing in the sugar, and finally the flour is very carefully folded in by hand.

Heavier types of cake such as gold cake, are usually mixed at low speed at first, *i.e.*, while the flour is being moistened, and at medium speed during the remainder of the process. The mixing is continued until maximum lightness is obtained, which takes from 10 to 15 minutes. During this time the batter has approximately doubled in size.

In making pound cake the speed of mixing has practically no effect on the quality of the finished product. The chief requirement is that the ingredients be thoroughly mixed.

### *Temperature of Mixing*

In making angel food cake, it is preferable to beat the egg white at a moderately low temperature, 50° to 55° F. A more tender cake results when the egg whites are beaten at this temperature. The ingredients in gold cake and pound cake should not be mixed at a temperature much above 70° F. When the temperature is too high *e.g.*, 90° F., much of the strength of the baking powder is dissipated. With too low a temperature the shortening congeals and a non-uniform mixture results. A temperature from 70° to 75° F. is preferable. When the ingredients are mixed at this temperature a larger cake with better texture is obtained.

### *Baking*

Baking a cake properly requires knowledge and experience. An oven with uniform and easily controlled heat should be used. The exact temperature best suited for baking the different kinds of cakes can best be determined by actual experience with the oven at hand. The kind of heat and the insulation and the load of the oven all

[4] Denton, Mina C., and Miller, Elizabeth W. Record of operations in cake making. University of Chicago, 1919. Mimeograph.

influence the temperature. Approximate temperatures can be quoted, but slight variation may be found necessary in individual ovens. Angel food cake may be baked at about 325° F., but gold cake and pound cake require higher temperature, *viz.*, approximately 375° F. For all kinds of cake most of the heat should be from the bottom. If there is too much top heat the cake will crust over, and the top will bake before the rest of the cake. The gas within the cake will then burst through the crusted top, leaving an unsightly break.

The time of baking depends upon the kind of cake and its size. Most cakes must be slowly baked at moderate temperature. Heat penetrates to the center of a cake very slowly, hence sufficient time must be allowed for the interior to reach approximately 210° F. or almost the boiling point of water before the cake is removed from the oven. If the temperature is too high the outside of the cake will scorch before the inside is thoroughly baked. To bake most cakes requires from three-quarters to one hour.

Owing to variations in temperature requirements of the different types of cake, it is best to bake only one kind of cake at a time in an oven. A cake should be baked at the maximum temperature that will not scorch it. Too little heat will dry the cake excessively, producing a coarse grain, a thick crust, and a crumbly cake. The temperature required to bake a lean cake mixture is higher than that required for a rich one. When thoroughly baked, a cake will draw away somewhat from the sides of the pan and settle slightly. To test whether the cake is sufficiently baked, it is only necessary to touch it lightly with the finger. If the cake is done, the dent made by the pressure of the finger will spring back; if not done the dent will remain, indicating that the heating should be continued a few minutes longer.

A cake that is under-baked will shrink excessively, cave in from the top, and have a moist dough-like area near the bottom. After baking, angel food cakes should be inverted and allowed to cool without being removed from the pan. Other cakes should be removed from the pan and cooled upon a rack.

### *Scoring*

Cakes may be "scored" or judged from two different points of view: (1) characteristics resulting from variations in constituents or methods of procedure, for example, the baked cake is used as a means of evaluating different flours, sugars, shortenings, etc., for cake making purposes; (2) characteristics resulting from the use of different formulas and different environmental conditions, for example, cakes from one bakery compared with cakes of the same class from a different bakery disregarding the constituents used or the conditions under which the

cakes were baked. Hence "score cards" used in judging cakes may vary somewhat, depending upon their purpose. For instance, one score card may enumerate depth of crust and color of crust. Both of these factors depend chiefly upon the constituents other than flour and the temperature and time of baking and not upon the quality or character of the flour being tested. In the same way a score card may assign a large part of the total score to odor and taste. These two items are likewise dependent upon the formula and are not due to any differences that may exist between two flours of normal characteristics.

In studying the effect of the character of flour as well as that of other ingredients upon the quality of cake, a special score card is needed. In our experiments all the cakes of a series were made by the same formula, only one ingredient being varied at a time, and they were baked for the same length of time and at the same temperature. In other words, the ingredient being studied was the only variable. A score card showing the differences, if any, caused by variations in the properties of the ingredients is desired. In such scoring the following points would be considered.

*Volume* depends to some extent on the type and quality of flour used, and hence should be considered when judging the suitability of flour for cake making. Large volume is not always desirable, as it may be associated with poor grain and texture. Large volume may also result from using too much leavening. On account of the coarse grain, such cakes dry out quickly.

*Lightness* or *specific gravity* is directly related to volume. A low specific gravity accompanies a large volume and indicates that the cake is light (not soggy). Lightness depends upon the proper formula, proper mixing and baking, and to a less extent upon flour characteristics.

*Shape* of the loaf may be influenced by the water absorption of the flour and by the quantity and quality of the gluten. The symmetry of the loaf as well as the character of the break in the top should be taken into consideration when scoring. A recessive top may indicate that an unbalanced formula such as too much sugar, shortening or leavening in proportion to the flour was used or that the cake was jarred before the structure became set, all of which will cause a cake to "fall." A sharply peaked top may indicate that the batter was too stiff, that is, too large a proportion of flour was used, or that there was too much top heat.

*Crust* differences are usually caused by variations in the baking conditions, but they may also be caused by variations in flour tolerances toward sugar, shortening, etc. Crust characteristics may be indicative of the temperature and time of baking or of the proportions

of certain constituents in the formulas. Thus, a thick crust may indicate too long a time in baking, and a sugary crust too large a proportion of sugar. However, a smooth and tender crust should be formed under proper conditions.

*Crumb* texture and grain are influenced by flour qualities; the texture very probably will be harsh and the grain coarse when a strong flour is used. The grain is also dependent upon the aeration of the batter and the evolution of carbon dioxide. But if the leavening is correct, variations in grain may be caused by differences in gluten quality. Also, too much leavening produces a coarse grain, and unequal distribution of the leavening will produce a nonuniform grain structure.

*Moistness* in the crumb depends to a large extent upon the formula and upon the method of baking, but it is also influenced somewhat by the character of the flour. A very soft flour produces a more moist cake than a strong hard flour.

*Tunnels* or large irregular holes in the crumb are due usually to incomplete mixing resulting in uneven distribution of the shortening.

*Color* in cake is affected by the presence of certain ingredients *e.g.*, egg yolk, spices, fruit, nuts, and chocolate. In a white cake, however, the color of the flour influences the color of the cake, and hence color may find a place on the score card.

*Flavor* or *taste* is dependent upon the constituents other than flour and their proportions and is not a characteristic attributable to the flour, assuming, of course, that the flour is normal. This factor then has no place on a score card to be used in scoring cakes from the standpoint of the suitability of flour. It does, however, have a very definite place on a score card used in judging cakes for quality. In fact, from a commercial standpoint, taste is one of the most important items on the whole score card. Any unpleasant or abnormal taste should be very adversely scored.

*Aroma* (*odor*) is another quality that is dependent upon the formula, and not upon the flour used. Although various constituents of the formula contribute toward the aroma of the finished cake, usually some substance is added to produce the aroma desired, and the effect is entirely independent of cake quality. However, in scoring cakes for their attractiveness, aroma must be given consideration. Any abnormal odor should be noted.

### *Analysis of Cake*

Besides scoring cake, which is mostly judging its visible and measurable physical characteristics, it is sometimes important to make a chemical analysis to determine its relative food value.

*Tabular statement of the various factors used in scoring cake:*

External
- General appearance—favorable or unfavorable
- Weight
- Volume—by seed displacement or by direct measurement (crude)
- Height—in inches or centimeters
- Shape—symmetry, break, rounded or receded
- Crust—thickness, tenderness, sugariness
- Color

Internal
- Grain
  - Cells—size and thickness; uniformity
  - Tunnels (lack of)
- Texture—tenderness, softness, smoothness, silkiness, moistness, resiliency
- Color
- Flavor (taste)
- Aroma (odor)

The methods employed for the chemical analysis of cake and its ingredients are those adopted by the Association of Official Agricultural Chemists (Official and Tentative Methods of Analysis, 3rd ed. 1930). The analysis may include the following determinations: Moisture, fat and fat constants, fiber, total nitrogen (water soluble N and alcohol soluble N), ash, sodium chloride in ash, ash constituents (calcium, magnesium, phosphorus, etc.), starch, lecithin and lecithin phosphoric acid, sugar, pH, acidity.

## Experimental Work on Cake Ingredients

### *Flour*

In this investigation three classes of flour were tested for their cake making properties, namely, hard spring wheat, hard winter wheat, and soft winter wheat flours. Three grades of each class were used, namely, patent, straight, and clear. In addition, each of these grades of flour was bolted into "fine" and "coarse" granulations [5] and each part was tested. Experiments were also conducted to test the effect on the quality of the cake of incorporating, with the flour, starch of different kinds, corn meal, corn flour, soybean flour, wheat germ, etc. The three types of cake were made with each of these combinations.

### *Class and Grade of Flour*

*Angel food cake.* In general a better angel food cake is obtained by using soft wheat flour than hard wheat flour, grade for grade. A hard wheat flour to which starch has been added in order to give it the protein content of a soft wheat flour may be used, but it does not make so good an angel food cake as the soft wheat flour itself. As a rule, the higher grades of flour, patent and straight, of both soft and

[5] The fine portion passed through the 20 or 25 XX silk bolting cloth; the coarse remained on the 14 or 15 XX.

hard wheat flours, are better suited for angel food cake than is a clear grade. With hard wheat flour, angel food cake made from the patent grade has a better grain, and greater uniformity of cell and texture and is more tender than one made from the straight grade, although the cake made from a straight grade often has a larger volume. With soft wheat flour there is little difference between the quality of angel food cake made from a patent grade and that made from a straight grade.

*Gold cake.* Gold cake showed no consistent and appreciable differences due either to the grade or the class of flour. This kind of cake is apparently not very sensitive to the variations, chemical and physical, of the different flours. The clear grade of some flours, especially from hard wheat, seemed to produce even better gold cake than did the patent or straight grade.

*Pound cake.* The use of flour of different classes made no appreciable difference in the quality of pound cake. Although, in general, a somewhat larger cake with better texture results from using the higher grades of flour, good pound cake may be made from a patent, straight, or clear grade of flour from either a moderately soft or a moderately hard wheat. The difference in the quality of pound cake resulting from the use of flour of different grades is less marked than is the difference in the quality of bread made from these flours.

### *Granulation or size of flour particles*

According to our experience, the fine portion of flour is better suited for cake than is the coarse portion. This is true irrespective of the class or grade of flour.

*Angel food cake.* Angel food cake made with the fine portion of flour was superior in size, grain, springiness, and appearance to that made from the coarse portion and even to that made from the original flour itself. Angel food cake made with the coarse portion of the flour of any grade is, however, more tender, but, in other respects, the poorest in quality.

*Gold cake.* The fine portion of flour, irrespective of class and grade, makes better gold cake than does the coarse part. The original flour likewise makes better cake than does the coarse portion. With the higher grades of flour, however, very good cake can be made from even the coarse granulates.

*Pound cake.* In pound cakes the fine portion generally produces a larger volume than does the coarse portion but in other respects there is no consistent difference in the quality of the cake, and this seems to apply irrespective of the class or grade of flour. In fact the flour as obtained from the mill makes as good a pound cake as does either

the fine portion or the coarse. In other words, good pound cake can be made from the original flour as well as from the fine and coarse particles of patent, straight, or clear grades of the average hard and soft wheat. Table XV records the composition of each separation of the soft and hard wheat flours tested.

TABLE XV

CHEMICAL ANALYSES OF GRANULATION SEPARATIONS OF FLOURS

| Class | Separations | Moisture | Ash | Protein (N×5.7) |
|---|---|---|---|---|
| | | % | % | % |
| Soft wheat, 1 | Original | 10.6 | 0.38 | 7.7 |
| | On 14 XX | 9.9 | 0.34 | 9.1 |
| | Thru 25 XX | 10.3 | 0.34 | 6.6 |
| Soft wheat, 2 | Original | 10.2 | 0.35 | 8.6 |
| | On 14 XX | 9.7 | 0.42 | 10.0 |
| | Thru 25 XX | 10.1 | 0.35 | 7.4 |
| Soft wheat, 3 | Original | 10.0 | 0.40 | 10.2 |
| | On 20 XX | 10.9 | 0.39 | 11.7 |
| | Thru 25 XX | 10.5 | 0.39 | 8.4 |
| Soft wheat, 4 | Original | 11.3 | 0.36 | 9.0 |
| | On 15 XX | 11.4 | 0.36 | 9.6 |
| | Thru 25 XX | 11.9 | 0.36 | 7.7 |
| Soft wheat, 5 | Original | 10.6 | 0.41 | 7.6 |
| | On 14 XX | 10.4 | 0.40 | 8.0 |
| | Thru 16 XX | 10.4 | 0.40 | 7.2 |
| Averages for flours, 1 to 5 | Original | 10.5 | 0.38 | 8.6 |
| | Coarse | 10.5 | 0.38 | 9.7 |
| | Fine | 10.6 | 0.37 | 7.5 |
| Hard spring wheat | Original | 12.4 | 0.38 | 12.4 |
| | On 15 XX | 12.6 | 0.38 | 12.7 |
| | Thru 25 XX | 12.3 | 0.42 | 10.9 |
| Hard spring wheat (special) | Original | 10.0 | 0.57 | 11.0 |
| | On 14 XX | 10.2 | 0.62 | 11.0 |
| | Thru 16 XX | 9.8 | 0.49 | 10.9 |
| Blend 50–50 hard spring and soft winter wheat | Original | — | 0.44 | 11.2 |
| | On 18 XX | — | 0.41 | 11.7 |
| | Thru 25 XX | — | 0.57 | 10.7 |

### *Use of Adjuncts in Cake Making*

Since cake contains too large a proportion of carbohydrates to be a good food when used as an important part of the diet, the substitution of high protein products, such as soybean flour and wheat germ, for part of the wheat flour would increase the protein and ash and reduce the carbohydrate content, thereby making the cake a more balanced food. Experiments were conducted to determine the effect

on the quality of the cake of incorporating the following substitutes, or adjuncts: corn-starch (both normal and gelatinized), starch from tapioca, rice, potato, wheat, and sago; corn flour, soybean flour, banana flour, wheat germ, whole wheat flour, potato flour, and artichoke flour.

*Angel food cake.* As a rule, the addition of 15 to 20% starch to any wheat flour of any grade improved the tenderness, size, texture, springiness, uniformity, and appearance of angel food cake. Tapioca starch, grade A, gave better results than did grade "K" or "Royal." Rice starch proved superior to all the other starches used.

With hard wheat flours even 30% starch can be satisfactorily used in making angel food cake.

In using hydrolyzed or partly gelatinized starch it is not advisable to add more than 15 to 20% on the basis of the flour, although good angel food cake resulted from the use of a somewhat larger amount of a few commercial hydrolyzed starches.

With hard wheat flour it was possible to incorporate as much as 30% of corn-flour, and from this mixture make a better angel food cake than with the wheat flour alone. With soft wheat flours, which are themselves specially suited for the making of angel food cake, it was not possible to use more than 10 to 15% of corn-flour and obtain an excellent cake, although the use of a larger amount produced a fairly good cake. Cake made from a mixture of corn-flour and wheat flour did not receive so high a rating as that made from a mixture of corn-starch and wheat flour. Corn-flour does, however, give to the cake distinctive characteristics, especially flavor, which may be enjoyed by those desiring a "different" angel food cake.

Soybean flour was used as an adjunct simply as a novelty. An occasional angel food cake made with a mixture of soybean flour and wheat flour was superior in tenderness and in some other ways, but in general this mixture cannot be recommended for making angel food cake.

Generally speaking, a more satisfactory cake of any type can be made with adjuncts when they are added to the higher grades of flour than to the lower grades and when used with strong flours rather than soft winter wheat flour.

While angel food cake is made with white flour, sugar, and eggs, it is quite possible to make an attractive angel food-like cake with whole wheat flour instead of white flour or with a mixture of white flour and soybean flour, wheat germ, or other products, especially when a novelty product is desired. The incorporation of any of these subsidiary flours will not only make a cake of peculiar outward characteristics but will increase the protein, mineral, and vitamin content of the

cake, all of which are present in ordinary angel food cake in small quantities only.

Angel food cake may also be made by replacing 5 to 10% of the soft wheat flour with banana powder. The banana powder imparts a pleasant flavor.

*Gold cake.* Gold cake is, as a rule, not improved by the use of starch of any kind with wheat flour. The same applies even more definitely to the use of the various corn-flours or soybean flour, though occasionally the cake made with 25% of these substitutes is superior in size, grain, springiness, or general appearance to that made with wheat flour only. The use of 20 to 25% of any of these adjuncts with wheat flour will make a good gold cake that is palatable though not of superior quality. The incorporation of a smaller amount, however, for example say 10 to 15%, does not affect the quality unfavorably to any appreciable extent, if at all.

The use of about 20% of wheat germ mixed with 80% white flour of any grade makes an attractive, nutty-flavored gold cake of very good quality. Whole wheat flour may also be used to make a novelty gold cake that will be enjoyed by those preferring products made from whole wheat.

Adjuncts can be used with better results with the stronger flours than with soft wheat flour.

*Pound cake.* A mixture of 20% of starch with 80% of a straight or clear grade of wheat flour sometimes makes a pound cake superior in appearance, uniformity of grain, springiness, and texture to one made from wheat flour alone. Rice starch and the better grades of tapioca starch are generally better suited for this purpose than is corn-starch. Gelatinized corn-starch is not so good as untreated starch as an adjunct to wheat flour for pound cake. No improvement was noted when treated or partially gelatinized starch was added, even to the extent of only 12.5%.

Better results are obtained when these starches are mixed with hard wheat flour than with flour made from soft wheat.

When any grade of hard spring wheat flour except patent is used for pound cake the addition of about 20% of corn-flour or corn-starch improves the appearance of the product as well as the texture and the uniformity of the grain. The addition of corn-flour or corn-starch to soft winter wheat flour, either patent or straight, does not improve pound cake. A straight grade of soft wheat flour to which corn-flour has been added makes a better pound cake than does a patent grade.

Almost without exception the addition of 20% soybean flour to the different grades of spring wheat flour resulted in a good pound cake though inferior in every respect to that made from wheat flour alone.

When soybean flour is used, better pound cake can be made with soft winter wheat flour than with the same grade of hard wheat flour. The advantage of soybean flour is its nutritive qualities, especially its high protein content, the quality of its protein, and its vitamin potency.

In making pound cake wheat germ can be used to the extent of 15 to 20% without injury to the quality of the cake. A larger proportion of germ makes a cake which, while very palatable, is not so attractive as when not more than 20% is used. Like soybean flour, wheat germ is rich in protein of good quality. Furthermore, it is rich in minerals, fat, and vitamins. The use of an appreciable amount of germ gives to the cake a "nutty" flavor and a greater food value. Pound cake can also be made with finely ground whole wheat flour instead of white flour.

Potato flour (not potato starch) when used to the extent of not more than 12.5% will make a good cake of the three types studied. The moisture content of the cake is slightly increased and the keeping quality improved as the result of using this small amount of potato flour.

Like potato flour, the flour made from the artichoke can be utilized in cake making. Our experience shows that good pound or gold cake can also be made from wheat flour to which 10 to 12% artichoke flour has been added.

### *Leavening*

*Effect on angel food cake.* In making angel food cake it is advisable to add sufficient acid ingredient (cream of tartar, calcium acid phosphate, or other acid reacting substance) to give to the finished cake a pH of approximately 5.0 to 6.0. The results of varying the pH are shown in Table XVI.

To secure the proper degree of acidity in an angel food cake batter, it is not necessary to use cream of tartar or calcium acid phosphate. The edible organic acids, such as citric, lactic, and acetic acids, may be substituted. These may be employed either as the pure concentrated acids or they may be used in their natural dilute forms, such as lemon juice, and vinegar. Approximately 1 ounce of lemon juice or vinegar is required to make an angel food cake which weighs 1 pound. If the concentrated organic acids are used, approximately the following amounts will be required: citric acid, 0.6 g.; lactic acid, 1.3 cc.; and acetic acid, 1.3 cc.

The use of lemon juice also serves other purposes. It facilitates whipping of the egg white to the proper consistency, and it noticeably improves the color of the cake. Furthermore, the quality of the cake in other respects is equal to that made with cream of tartar.

TABLE XVI

pH OF ANGEL FOOD BATTER AND CAKE[1]

(Based upon use of 90 g. of egg white)

| Cream of tartar | pH | | Character of cake | Calcium acid phosphate | pH | | Character of cake |
|---|---|---|---|---|---|---|---|
| | Batter | Cake | | | Batter | Cake | |
| *Grams* | | | | *Grams* | | | |
| 0 | 7.6 | 8.4 | Fair, dark crumb | 0.0 | 7.8 | 7.3 | Fair, dark crumb |
| 0.4 | 7.5 | 7.9 | Good | 0.5 | 7.9 | 6.5 | Good |
| 0.8 | 7.1 | 7.1 | " | 1.0 | 6.2 | 5.9 | " |
| 1.2 | 6.3 | 6.3 | Very good | 1.5 | 5.7 | 5.4 | " |
| 1.6 | 5.4 | 5.7 | " " | 2.0 | 5.1 | 4.9 | " |
| 2.0 | 5.1 | 5.4 | Excellent | 2.5 | 4.9 | 4.5 | Good (acid flavor) |
| 2.4 | — | 5.3 | " | 3.0 | 4.7 | 4.3 | Good (acid flavor) |
| 2.8 | — | 5.0 | " | — | | | |
| 3.2 | — | 4.9 | Excellent (acid flavor) | | | | |

[1] The pH was determined electrometrically by T. L. Swenson, U. S. Bureau of Chemistry and Soils.

Experiments in which the amount of acid ingredient was varied showed that for a one-pound angel food cake (made by using 3 oz. of flour), the best results were obtained with approximately 3.5 g. of calcium acid phosphate or 4 g. of cream of tartar. Larger amounts of these acid ingredients produced angel food cake of very good appearance but which was too acid in taste. The use of smaller amounts make cake inferior in texture, grain, and appearance. When no acid ingredient at all was used, the cake was dark in color, and otherwise inferior.

*Effect on gold cake.* In making gold cake it is immaterial whether a tartrate, phosphate, combination, or other baking powder is used so long as it is not added until near the end of the mixing period. A slight increase in the amount of baking powder used does not affect the cake appreciably, though it is advisable not to vary much from the amount given in the formula (see p. 191). To make good gold cake a leavening agent must be used. A tendency of scgginess may sometimes be averted by slightly increasing the amount of baking powder. About 4 to 4.5 g. of baking powder should be used per pound of finished gold cake, which requires approximately 125 g. (4⅓ ounces) of flour.

*Effect on pound cake.* In making pound cake the use of leavening is not essential, though our results show that the addition of a small

amount of baking powder not only increases the size of the cake, but improves its texture, uniformity of grain, and general appearance as well. To make one pound of pound cake requires only one-third as much baking powder as is used for gold cake.

### *Eggs*

*Use in angel food cake.* For angel food cake, fresh or frozen egg whites are superior in foaming qualities to the average commercial dried egg white, when whipped with cream of tartar or calcium acid phosphate, or other acid ingredients. One very important point noted in the use of egg white, both from fresh eggs and from eggs that had been stored for nine months, was that the thin portion made a better cake than the more viscous albumin. It was easier to whip the thin portion to the proper consistency. Apparently air is more easily incorporated into the thin or less viscous whites. It was further noted that when enough water to make the viscosity comparable to that of thin egg white was added to the thick or viscous portion of the whites an improved angel food cake was obtained. It is of the utmost importance therefore that in all investigations in which egg white is used, either a separation be made of the thin and thick whites and each used separately or that a uniform mixture of the two kinds of whites be made. These results confirm those obtained by Hunt and St. John,[6] who demonstrated that adding water to the thick portion of egg white improves its whipping qualities.

Our experiments have shown that the quantity of egg white in an angel food cake recipe can be decreased by one-third, if the difference in volume is made up with water. Increasing the egg white by one-third, however, did not result in a satisfactory cake from the standpoint of appearance.

*Use in pound cake.* In making pound cake, increasing the amount of egg in the laboratory formula (p. 191) from one-third to one-half somewhat improved the grain and texture, but not the size or general appearance. Decreasing the amount of egg from one-third to one-half made the cake soggy or "sad," with a smaller volume, and poorer grain, texture, and general appearance. If the amount of egg is decreased, as it is in many commercial pound cakes, it is necessary to increase somewhat the amount of both fat and milk to make a satisfactory product.

*Use in gold cake.* Experiments in making gold cake with dried yolk showed very clearly how ill-adapted this product is for cake making. The cake made with dried egg yolk was smaller, and had a

[6] Hunt, Leila Wall, and St. John, J. L. Angel food cake from the thick and thin portions of egg white. J. Home Econ. 23: 1151–6 (1931).

less attractive appearance, and poorer texture and grain than that made from fresh yolk. The grain was very irregular, large holes predominating, due to the difficulty of properly reconstituting the dried yolk into a smooth uniform mass free from lumps.

Because of the difficulty of reconstituting the dried whole egg into a lump-free liquid it is likewise better to use fresh or frozen eggs for pound cake.

*Effect of Varying the Amount of Liquid*

Experiments were made with gold cakes in which the milk content varied by 5 cc. increments from 15 to 70 cc. per cake. The regular formula calls for 44 cc.

Cakes made with greatly reduced quantities of milk were of smaller volume, and poorer in grain, texture, and appearance. They were peaked in the center of the top.

When greatly increased quantities of milk were used, the cakes were flat on top. They had poor volume, grain, texture, and appearance. Some of the cakes containing the most milk had a tendency toward sogginess. The loss of weight in baking is almost the same regardless of the percentage of milk in the batter. Thus, the loss in weight ranged irregularly from 19 to 25 g. while the milk content ranged regularly from 15 to 70 cc. From these results it is evident that the moisture content retains practically the same ratio in the cake as in the batter, *i.e.*, the more moisture in a batter, the more it will be retained in the finished cake.

Some commercial cakes are made with dried egg white and dried milk powder, which must be reconstituted by the addition of water. In making a special type of white cake according to the formula given on page 191, the best product was obtained by using approximately 270 cc. of water to dissolve the egg white and milk powder. A decrease in the amount of water by 20%, or to about 215 cc., resulted in a cake of practically the same quality as when 270 cc. of water was used, but if the water was decreased to 200 cc., the quality of the cake was decidedly inferior.

*Shortening*

Vegetable shortenings produce just as attractive appearing cakes as does butter though it has generally been found that the latter will give a better flavor and more tenderness. This applies to both gold cake and pound cake, but more especially to the latter because of the larger amount of fat used.

*Effect on angel food cake.* In making angel food cake every precaution is taken to avoid the inclusion of even a trace of yolk in the egg white, on the grounds that the fat in the yolk will lower the surface

tension of the whites, making it difficult to beat them into a foam of proper consistency. Even the oil used as a flavor impairs somewhat the quality of the cake, making it almost impossible to produce the highest class product.

It has often been stated not only that there should be no trace of fat in the egg white, but that the pan in which it is baked must be absolutely free from grease. According to our experiments no marked difference in the quality of the cake resulted from lightly greasing the pan.

*Effect on gold cake.* Increasing the fat over the amount specified produced a gold cake of better volume, appearance, and texture. The grain of the cake was essentially as good, but the uniformity of grain was slightly inferior. Decreasing the fat one-half or one-third resulted in a cake of lower volume and poorer appearance, texture, and grain. The uniformity of grain, however, was slightly improved.

### *Sugar*

Ordinary granulated sugar was separated into several portions according to the size of the crystals and tested with the three types of cake. It was found that sugar fine enough to pass through a 30 or 40 mesh sieve made better cakes than that which passed through a 20 mesh. "Fruit" or berry sugar is the commercial term for the finely granulated product.

*Effect on angel food cake.* In making angel food cake better results were obtained by using either all powdered sugar or even a mixture of 3 parts of fine granulated and 1 part of powdered than by using all granulated sugar.

*Effect on pound cake.* Increasing the sugar from one-third to one-half over the amount specified in our laboratory formula for pound cake (p. 191) appreciably improved the cake in all respects, and made the grain of the cake similar to that of gold cake. Decreasing the amount of sugar by one-third produced a cake much inferior in all respects but especially in grain and uniformity of cell.

In making all three types of cake, commercial brands of invert sugar found on the market were substituted for cane sugar to the extent of 5, 10, and 15%, with quite satisfactory results. In many of our experiments with invert sugars the angel food cake was quite as good as that made with cane sugar alone, and the gold cake was even better. The use of a small amount of invert sugar keeps cake fresh for a longer period.

### *Flavoring*

In conjunction with the Bureau of Chemistry and Soils' activity on the utilization of citrus by-products, experiments were carried on

to determine whether dried orange peel (a waste product) could be used as a flavor for gold cake and pound cake. Cakes were made with orange peel, concentrated orange juice, orange oil, and lemon juice. The orange peel was very satisfactory and seemed to give the cake excellent keeping qualities. Orange oil proved to be a most delightful and satisfactory flavor, but the concentrated orange juice was not so satisfactory. Lemon juice can, of course, be used with perfect satisfaction for the three types of cake.

### *Salt*

*Effect on angel food cake.* Doubling the amount of salt specified in our formula (p. 191) yielded angel food cake with improved appearance but with somewhat too acid a taste. One gram of salt is sufficient for an angel food cake weighing 1 pound; when 2 g. of salt was used the acid taste was too pronounced.

*Effect on gold cake.* It was quite possible to use as little as 0.75 g. salt or as much as 1.5 g. for each pound of gold cake without noticeably affecting the taste.

*Effect on pound cake.* The amount of salt may be varied considerably without any appreciable effect upon the size, grain, texture, or general appearance of pound cake. Whether 0.75 g., 1.25 g., or 2.05 g. of salt was used per pound of cake there was little difference in the quality of the product.

### *Moisture-retaining agents*

Glycerine is sometimes used by bakers for the purpose of retaining moisture in cake. Experiments in which a very small amount of glycerine (1 to 2 g.) per pound of cake was used proved more efficient in this respect than did larger amounts, *i.e.*, 10 g. Hydrolyzed starch or invert sugar is used for the same purpose.

## Summary

In conducting these experiments hundreds of cakes were made. The results herein recorded represent the predominant tendencies. Undoubtedly with ingredients having different characteristics, distinctly different results would be obtained.

Soft wheat flour (patent grade) is preferable for cake making. A flour with weak gluten is best for the foam type of cake. For the heavy cakes, the flour should be moderately strong.

Soft winter wheat flours, grade for grade, yielded better angel food cake than did the hard spring wheat or hard winter wheat flours.

Soft winter wheat flour made better angel food cake than did a strong flour to which sufficient starch had been added to reduce the gluten content to that of the weak flour.

The higher grades of flour, patent and straight, of any class are better suited for making angel food cake than are the clear grades.

Gold cake was not so appreciably affected as was angel food cake by the class or grade of flour.

Pound cake of good quality was made from all the recognized flour grades from both the hard and the soft wheats and blends of the two. A flour of medium strength was the best.

The finer particles of flour are better suited for angel food cake and gold cake than are the coarse particles. This applies to all grades and classes of flour.

The finer particles of soft wheat flour made better cake of all types than the corresponding size granulates of hard wheat flour.

Good pound cake was made from both the fine and coarse granulates of patent, straight, and clear grades of flour from both hard and soft wheats.

Excellent angel food cake was obtained with wheat flour to which 15 to 20% starch or corn flour had been added. Even 25 to 30% of starch or corn flour can be used successfully with hard wheat flours. Banana powder to the extent of 5 to 10% of the weight of the flour also gave good results.

In cake of the three types hydrolyzed or partly gelatinized starch can not be used to the same extent as ordinary starch.

Good gold cake and pound cake were made with mixtures of 20% soybean flour and 80% wheat flour.

More satisfactory cakes of all types were made when the adjuncts were added to the higher grades of flour and when the flour used was from hard wheat rather than from soft wheat.

Whole wheat flour gave good results in cake-making.

A mixture of 20% wheat germ and 80% flour made very palatable gold cake and pound cake.

The best angel food cake is made when the pH of the latter is in the range of 5.0 to 6.0. The acid used may be cream of tartar, acid calcium phosphate, lemon juice, vinegar, or dilute citric, acetic, or lactic acid. Excellent angel food cake was made by using lemon juice as the acid ingredient.

Gold cake and pound cake are improved by the addition of leavening agents. Even pound cake is better if a small amount of leavening is used. A relatively large quantity is required for gold cake. Only sufficient leavening to make a cake light should be used. An excess produces a coarse grained cake that dries out and becomes stale quickly.

Both fresh and frozen liquid eggs make satisfactory cakes of all types.

Dried egg white made a fairly good angel food cake. The cake made with fresh or frozen egg white, however, was of far better quality.

Dried egg yolk and dried whole egg can not satisfactorily replace the fresh egg or egg yolk for making gold or pound cake.

Oil or fat, even traces, should be excluded from angel food cake batter, but the pan may be lightly greased without influencing the cake to any great extent.

Increasing the specified fat content of gold cake by one-third improved the cake. Decreasing the fat by one-third resulted in an inferior cake.

Coarse sugar is not suitable for cake making. Either very fine granulated sugar (berry or fruit sugar) or powdered sugar gives better results.

Increasing the sugar content of pound cake by one-third improved it appreciably, particularly the texture and grain.

Orange peel and orange oil are satisfactory flavors for gold cake and pound cake.

An appreciable excess of salt in angel food cake brings out too prominently the acid taste due to the presence of cream of tartar, or other acid component.

Cake made with small amounts of glycerine, invert sugar, or hydrolyzed starch had somewhat better keeping qualities than cake in which these substances were not used.

The "single phase" method of mixing the ingredients produced as good a cake as the "creaming" method.

The production of a fat-in-water combination during the creaming process of mixing, resulting in a curdled mass, had no detrimental effect on the quality of the cake.

# THE OCCURRENCE OF ACETYLMETHYLCARBINOL IN BREAD AND ITS RELATION TO BREAD FLAVOR

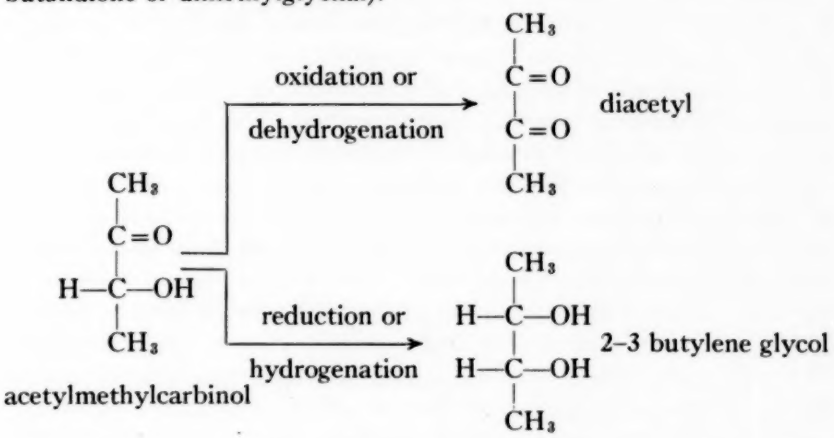
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(Presented at the Annual Meeting, June 1934)

## Introduction

*Acetylmethylcarbinol* (acetoin, dimethylketol, methyl 1-hydroxyethyl ketone or 2-butanolone-3) is closely related to *2–3 butylene glycol* (butandiol-2 : 3, 2 : 3 dihydroxybutane, or dimethyl ethylene glycol) and also to *diacetyl* (dimethyl diketone, 2 : 3-diketobutane, butandione or dimethylglyoxal).




All three compounds are found as natural constituents of various food products and are among the products of the metabolism of various micro-organisms.

Acetylmethylcarbinol and 2–3 butylene glycol were first found by Harden and Walpole (1906) among the products of the fermentation of sugar by *B. lactis aerogenes*. Lemoigne and later Kluyver, Donker, and Visser't Hooft (1925) found these compounds among the products of the metabolism of various other micro-organisms, including yeasts. For a discussion of the earlier literature refer to Kluyver, Donker, and Visser't Hooft (1925) who modified Lemoigne's method for the determination of acetylmethylcarbinol and 2–3 butylene glycol and who showed that addition of hydrogen acceptors such as, *e.g.*, oxygen to fermenting sugar solutions, leads to formation of these compounds.

Visser't Hooft (1925) used the presence of acetylmethylcarbinol as a measure of the quality of those vinegars which are made by biochemical oxidation from fermented fruit juices. Van Niel (1927) further improved the method for the determination of acetylmethylcarbinol and the related compound diacetyl. Neuberg and Reinfurth (1923) found formation of acetylmethylcarbinol when acetaldehyde was added to sugar solutions fermented by yeast. Neuberg accepts the enzyme "carboligase" as responsible for the condensation of acetaldehyde to acetylmethylcarbinol. Van Niel, Kluyver, and Derx (1929) found that diacetyl is the principal constituent of the aroma of butter. Neither 2–3 butylene glycol nor acetylmethylcarbinol has any odor of its own. Any odor which has been attributed to acetylmethylcarbinol must have been due to traces of diacetyl present. Schmalfuss and Barthmeyer (1929) found diacetyl as an aroma constituent in tobacco smoke, roasted coffee, cocoa butter, beer, and honey. They did not find any diacetyl in tea, candy, and bread. Lemoigne and Monguillon (1930) found traces of acetylmethylcarbinol and 2–3 butylene glycol present in germinating grains. Gerhardt (1934) made a critical survey of the literature on diacetyl and butter aroma. Michaelian, Farmer, and Hammer (1933) studied the relationship of acetylmethylcarbinol and diacetyl to butter cultures.

The literature on the *flavor of bread* is very limited. Most of what is written on the flavor of bread has been a matter of opinion and very few scientific facts are recorded. In the first place there seems to be a wide divergence of opinion on what constitutes flavor. In our work the word flavor is used to indicate the combination of odor and taste. Substances which only affect the sense of taste by creating a sensation of sweetness, saltiness, bitterness, or sourness are, strictly speaking, not flavoring materials if they do not, at the same time, affect the sense of odor. It is not thought that the sensations of odor and taste can be separated when eating bread and therefore the term flavor in its above sense seems more appropriate to indicate the sensation which we experience when tasting and at the same time smelling bread.

Flavor of bread has been attributed by various authors to acetic acid, butyric acid, propyl alcohol, butyl alcohol, amyl alcohol, volatile ethyl esters, and other esters formed from these acids and alcohols, but, as stated before, no scientific evidence is presented. Summarizing the literature on bread baking it seems the consensus of opinion that flavor of bread is caused by two factors. The first factor is a blend of the flavors of the various ingredients. The second factor is the flavor produced during the fermentation as a by-product of the formation of alcohol and carbon dioxide. This last factor is very much dependent on the way in which the fermentation has been conducted.

In recent years there has been a tendency to blame modern methods and modern machinery for the decrease in bread consumption because of a supposed inferiority in flavor of modern bread compared with the old-fashioned, home-made bread. Quinn (1933) states the problem as follows: "We have better yeast, better flour, more hygienic bakeries, up-to-date machinery and ovens, greater scientific knowledge, better looking bread in many ways, and yet, on the whole, unsatisfactory bread. Why is it?" One reason seems to be lack of a proper method to evaluate the all important flavor of bread. Platt (1931) has reviewed the great difficulties which are encountered in the flavor scoring of food products by the ordinary organoleptic methods. He gives various excellent directions for the scoring of flavor of food products and states that such scoring is still the only way to express many important qualities of food products.

In this article is described a new chemical method with which an attempt is made to chemically evaluate differences in flavor between different bread samples. This method will not replace the organoleptic testing methods which are, of course, of paramount importance. It may be used in addition to the old organoleptic scoring methods in research and control work and may result in drawing renewed attention to the importance of flavor from the consumers standpoint.

The direct cause of the investigations reported here was an observation which one of the authors (Visser't Hooft) made when visiting bakeries. It was thought that the pleasant aroma which can be found around bakeries was somewhat similar to the aroma of good vinegar and also reminded one somewhat of the flavor of butter. This observation, together with previous work done by Visser't Hooft (1925), led us to the investigation of the problem as to whether acetylmethylcarbinol or diacetyl was in any way connected with the flavor of bread.

## Method for Determination of Acetylmethylcarbinol in Bread and Dough

Several loaves of commercial bread were tested for the presence of acetylmethylcarbinol or diacetyl. To our pleasant surprise acetylmethylcarbinol was found in many commercial loaves of bread which were considered to have good flavor and no acetylmethylcarbinol was found in other loaves which were thought to have a poor flavor. There was no diacetyl in any of these loaves.

This discovery of a heretofore unknown ingredient in ordinary bread was sufficiently important to warrant further investigations. A procedure for the determination of acetylmethylcarbinol in bread and dough was worked out. A number of tests were made and a

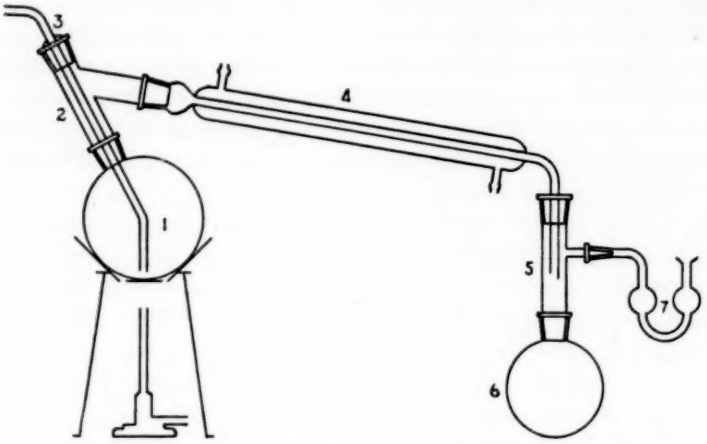



Fig. 1. Apparatus for the determination of acetylmethylcarbinol in bread and dough.

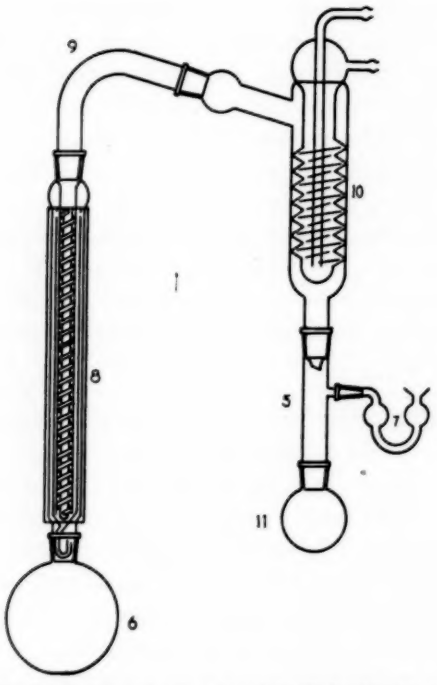



Fig. 2. Apparatus for the determination of acetylmethylcarbinol in bread and doughs.

procedure which is based on the methods as described in the literature previously cited was finally adopted. The principle of this method is the oxidation of acetylmethylcarbinol to diacetyl and determination of diacetyl after distillation as nickeldimethylglyoxim.

### *Description of Apparatus Used*[1]

The apparatus used is illustrated in Figures 1 and 2 consisted of the following parts: (1) 2000 cc. round-bottom flask; (2) three-way connecting tube; (3) steam inlet tube; (4) Liebig condenser; (5) three-way connecting tube; (6) 1000 cc. round-bottom flask; (7) gas trap; (8) 12″ Widmer column; (9) connecting tube; (10) Friedrich glass screw condenser; (11) 100 cc. round-bottom flask. All connections were standard interchangeable ground glass joints.

### *Reagents Used*

Ferric oxide.
Concentrated sulfuric acid.
20% solution of nickelous chloride in water.
20% solution of sodium acetate in water.
20% solution of hydroxylamine hydrochloride in water.

### *Procedure*

In a round-bottom flask (1) place 8 g. of ferric oxide, 400 cc. distilled water and 10 cc. concentrated sulfuric acid. To this mixture add 150 g. of the finely divided bread crumb. Connect the flask with the apparatus as sketched in Figure 1. Fill the trap (7) with about 2 cc. of a freshly made nickel reagent mixture consisting of:

20 cc. nickelous chloride solution
30 cc. hydroxylamine hydrochloride solution
50 cc. sodium acetate solution

The mixture in the flask is heated gently by leading in steam through inlet tube (3) and by a small flame under flask (1). Gentle heating is continued until the bread has disintegrated and the mixture stops foaming. The heat is then increased and 500 cc. of steam distillate is collected in flask (6). This distillation should take about 1½ hours. To the distillate in flask (6) add: 8 g. ferric oxide and 10 cc. concentrated $H_2SO_4$ and connect the flask with the distillation column of the apparatus as sketched in Figure 2. In the receiving flask (10), of this apparatus, place 10 cc. of the nickel reagent made up as described above. The trap (7) of the first apparatus is also transferred to the second apparatus. The flask (6) is heated and the

[1] Apparatus was furnished according to our specifications by Scientific Glass Apparatus Co., Bloomfield, N. J.

diacetyl formed is distilled over very slowly until the distillate reaches a 40 cc. mark on the receiver (30 cc. distillate collected). If an appreciable quantity of acetylmethylcarbinol was present in the original sample, a red precipitate will now be observed. The nickel reagent from the trap (7) is added to the mixture in flask (11), and the flask tightly stoppered with a rubber stopper which is tied on securely. The flask (11) is immersed for 30 minutes in boiling water. The precipitate is filtered off onto a tared Schott glass filter and washed with 100 cc. boiling water, dried in oven, washed with 10 cc. petroleum ether, and dried to constant weight at 110° C. Washing with petroleum ether is necessary to eliminate the small amounts of fatty material which distill over. The weight of the nickeldimethylglyoxim found, multiplied by the factor 0.609 gives the weight of the acetylmethylcarbinol present in the original sample of bread.

The analysis of a dough is carried out in the same manner.

The amounts of acetylmethylcarbinol found in the tests ran from 0 to 18 mg. of acetylmethylcarbinol per 150 g. of bread crumb. Check tests on this procedure were made by adding various known amounts of acetylmethylcarbinol to bread samples which were then analyzed. Approximately 80% of the acetylmethylcarbinol was recovered as nickeldimethylglyoxim. Many duplicate tests were made which indicate an experimental error of $\pm$ 0.5 mg. The nature of the material and the many manipulations and distillations which are necessary, do not permit a higher degree of accuracy in determining these very small quantities of acetylmethylcarbinol.

The red precipitate was identified as nickeldimethylglyoxim by liberating the dimethylglyoxim which was identified by its melting point. A melting point of 234° C. was found for the product derived from bread, 237° C. for the product obtained from Eastman Kodak Co., while the melting point of the two products mixed was found at 235° C. These tests prove that the substance found in bread was acetylmethylcarbinol and not one of its homologues.

### Determination of Acetylmethylcarbinol Figure "A.F." of Commercial Loaves

The term "acetylmethylcarbinol figure" or "A.F." is used to indicate the number of milligrams of acetylmethylcarbinol which were found in 150 g. of a certain bread dough or in 150 g. of the crumb of a certain loaf of bread. Although our tests showed that the crust of a loaf of bread contains acetylmethylcarbinol, the major portion of the acetylmethylcarbinol is found in the crumb and therefore in our A.F. determinations we used the bread crumb to get comparative results.

The following table gives some of the results which were obtained in determining the A.F. of various samples of commercial bread.

TABLE I

ACETYLMETHYLCARBINOL FIGURE FOR COMMERCIAL BREADS

| Brand | A.F. | Brand | A.F. | Brand | A.F. | Brand | A.F. |
|---|---|---|---|---|---|---|---|
| 1 | 0.0 | 4 | 2.4 | 7 | 5.3 | 10 | 10.4 |
| 2 | 0.9 | 5 | 2.5 | 8 | 6.5 | 11 | 15.0 |
| 3 | 2.1 | 6 | 4.2 | 9 | 7.6 | | |

Taking into consideration the fact that only 80% of the acetylmethylcarbinol present can be found, evidence is present that in an ordinary commercial pound loaf of bread about 30 to 60 mg. of acetylmethylcarbinol is frequently present. Acetylmethylcarbinol, itself odorless, slowly oxidizes to diacetyl with its pronounced odor and the presence of 30 to 60 mg. of acetylmethylcarbinol in a loaf of bread should therefore be of great importance as a flavor factor. To prove this a large room was sprayed with a quantity of diacetyl, equivalent to the amount of acetylmethylcarbinol in one loaf of bread, and this was sufficient to produce an unmistakable odor. We will refer to this question of bread flavor again later.

After testing a number of commercial breads some of the breads baked in our own laboratory were tested and to our surprise no trace of acetylmethylcarbinol could be found in loaves baked according to our usual standard baking formula. We will refer to this point again and show how conditions were changed so as to produce, in the laboratory, loaves with a good A.F.

Before these tests are described we want to discuss the origin of the acetylmethylcarbinol in bread and present a scheme which explains its formation. This scheme will be helpful in understanding our further research on the factors which control the A.F. of bread.

## Origin of Acetylmethylcarbinol in Bread and Scheme Showing Its Formation

None of the normal constitutents of a bread dough contain either 2–3 butylene glycol, acetylmethylcarbinol, or diacetyl. The only exceptions might be yeast and butter which sometimes contain traces. These traces are so small, however, that they cannot explain the A.F.'s which we have found. *Acetylmethylcarbinol is formed in the bread dough by the yeast during the fermentation period as a by-product of the alcoholic fermentation of sugar.* We have followed this formation in the dough by the determination of the A.F. at different points in a

number of different tests. Under certain conditions the acetylmethylcarbinol which has been formed during the early stages of the fermentation disappears again later. In those cases it is reduced to 2–3 butylene glycol. In normal breads the quantities of 2–3 butylene glycol are so small that they cannot be found by our present analytical methods. We succeeded, however, in proving the presence of 2–3 butylene glycol in straight doughs to which we had added acetylmethylcarbinol. 2–3 butylene glycol is not easily oxidized and it is not liable to oxidation in the bread; it has no odor of its own and therefore any acetylmethylcarbinol which has been reduced to 2–3 butylene glycol is lost. As stated above, the odorless acetylmethylcarbinol itself oxidizes slowly to diacetyl with its strong odor and so plays an important part in the flavor of the bread. A delicate balance between these various oxidations and reductions in the bread dough seems to be necessary to produce the most favorable results as far as flavor is concerned.

To further clarify these oxido-reductions we present the following scheme of the alcoholic fermentation of glucose. The chief result of this fermentation is represented by the classical equation:

$$C_6H_{12}O_6 = 2CO_2 + 2C_2H_5OH$$

According to the latest fermentation theories of Harden, Neuberg, Kluyver, and other investigators, methylglyoxal hydrate is an intermediate in this fermentation. For a clear discussion of these theories we refer to Kluyver (1931).. The intermediate methylglyoxal hydrate is subject to the following oxido-reductions:

$$\text{I.}\quad CH_3-CO-C(H)(OH)-OH + CH_3-CHO \quad \text{(acetaldehyde)}$$

(methylglyoxal hydrate)

$$= CH_3-CO-COOH + CH_3CH_2OH$$

(pyruvic acid; ethyl alcohol)

$$\text{II.}\quad CH_3-CO-COOH = CH_3-CHO + CO_2$$

(pyruvic acid; acetaldehyde; carbon dioxide)

In reaction I, acetaldehyde acts as a hydrogen acceptor and can be replaced by other hydrogen acceptors. Hence, in the presence of other hydrogen acceptors there will be an accumulation of acetaldehyde in reaction II. When such an accumulation of acetaldehyde occurs in the presence of yeast, the yeast will exert its so-called "carboligase" function and reaction III occurs:

$$\text{III.}\quad 2CH_3-CHO = CH_3-CO-CHOH-CH_3$$

(acetaldehyde; acetylmethylcarbinol)

In bread dough reactions I and II occur regularly and lead to the formation of ethyl alcohol and carbon dioxide. Reaction III only occurs when there are sufficient other hydrogen acceptors present to take the place of acetaldehyde in reaction I. Acetylmethylcarbinol once formed in the dough according to reaction III can disappear again either through oxidation (dehydrogenation) or through reduction (hydrogenation).

IV. Oxidation (dehydrogenation):

$$\underset{\text{acetylmethylcarbinol}}{CH_3-CO-CHOH-CH_3} + \underset{\text{H-acceptor}}{O} = \underset{\text{diacetyl}}{CH_3-CO-CO-CH_3} + H_2O$$

V. Reduction (hydrogenation):

$$\underset{\text{acetylmethylcarbinol}}{CH_3-CO-CHOH-CH_3} + \underset{\text{active hydrogen}}{2H} = \underset{\text{2–3 butylene glycol}}{CH_3-CHOH-CHOH-CH_3}$$

In the dough, in the presence of yeast, there is a tendency toward reduction according to reaction V. Reaction IV usually does not occur in the dough. Even if it did occur the diacetyl produced would be lost during the baking because diacetyl has a very low boiling point (86° to 90° C.). On the other hand, if reaction V can be controlled or eliminated in the dough, acetylmethylcarbinol can be kept in the finished bread because it will not disappear during the baking. Acetylmethylcarbinol has a boiling point of 140° to 144° C. In the finished bread reaction IV will probably occur slowly. Traces of diacetyl will be formed which will add to the flavor of the loaf.

From the above scheme it will be clear that in order to obtain this flavoring effect in bread, care must be taken to favor the formation of acetylmethylcarbinol according to reaction III and disfavor the undesirable reduction according to reaction V. By determining the A.F. we have a measure of this flavoring effect.

## Factors Influencing the A.F. (Acetylmethylcarbinol Figure) of Dough and Bread

It is a well established fact that the choice of ingredients as well as the baking procedure determine the characteristic flavor of yeast-leavened bread.

In our experiments it was found that the main factors which favor the formation of acetylmethylcarbinol in the dough and disfavor its reduction are the following:

1. High sugar-yeast ratio.
2. Diastase preparations.
3. Low fermentation temperature.
4. Short fermentation time.
5. Sponge-dough method.
6. Hydrogen acceptors or oxidizing agents and oxygen.
7. Proper yeast selection.

An appropriate combination of these factors will, of course, give the most desirable results and will have to be worked out for each individual case where a high A.F. is desired.

The following graphs indicate tendencies and were drawn from results obtained in a number of experiments made to determine the effect of the above individual factors. As much as possible, in each series of experiments, only one factor was varied at the time.

### 1. *Influence of Sugar-Yeast Ratio*

Experimental doughs of the following formula were made up with amounts of sugar varying from 0 to 5% (0 g. to 17.5 g.): Flour 350 g., water 200 cc., yeast 10 g., shortening 8 g., salt 6 g. These doughs were fermented at 86° F., and samples were taken at regular intervals for determination of A.F. The results of these tests are presented in Figure 3. An increase in sugar from 0 to 5% increases the A.F.

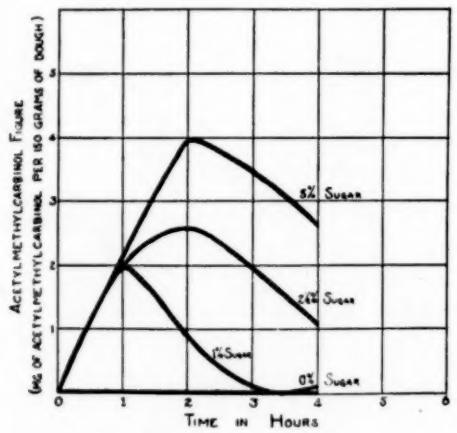



Fig. 3. Influence of sugar-yeast ratio.

maximum of a dough and also leaves the dough with higher A.F. at the time that the dough is ripe.

### 2. *Influence of Diastase Preparations*

Diastase preparations increase the available sugar in the dough and our experiments showed that in accordance with our tests on sugar-yeast ratio, as expected, diastase preparations increase the maximum A.F. of a dough and the A.F. of the ripe dough.

### 3. *Influence of Fermentation Temperature*

Low fermentation temperature is favorable for the formation of acetylmethylcarbinol. Doughs of the same composition as given under 1 were fermented at temperatures of 77° F., 86° F., and 96° F. The amount of sugar used was 2.5% (8.7 g.) in all cases. The results of these tests are given in Figure 4.

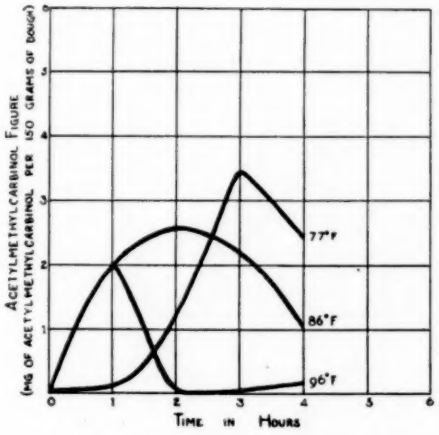



Fig. 4. Influence of fermentation temperature.

A low fermentation temperature causes a slower formation of acetylmethylcarbinol but a larger quantity remains in the dough after a fermentation time of 4 hours.

At higher fermentation temperatures the acetylmethylcarbinol is formed faster and disappears also faster, resulting in a low A.F. after 4 hours.

### 4. *Effect of short Fermentation Time*

A study of Figures 3 and 4 will show that the peak of the A.F. of a straight dough is reached before the dough is ripe. The fermentation time should, therefore, be kept as short as possible to retain as much acetylmethylcarbinol as possible at the time the dough goes to the oven.

### 5. *Influence of Doughing Procedure*

The sponge-dough method is more favorable for the formation and retention of acetylmethylcarbinol in a dough than the straight-dough method. In our comparative experiments we used the same formula as given above under 1 (with 3% sugar) for the straight dough. The sponge in the sponge-dough method was made up with 200 g. of flour, 55% of the total water, and 10 g. of yeast. The other ingredients were added when the sponge was mixed into a dough. The fermentation temperature in both cases was 80° to 82° F. Doughs were proofed for 1 hour at 90° F. The results of these tests are shown in Figure 5.

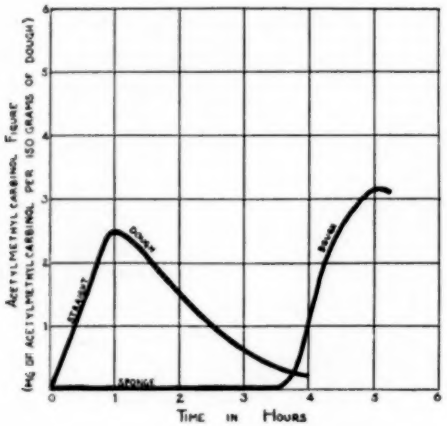



Fig. 5. Straight-dough and sponge-dough method.

In the straight-dough method the maximum A.F. occurs after one hour fermentation. Thereafter the A.F. drops and there is practically no acetylmethylcarbinol left after proofing when the dough goes to the oven. In the sponge-dough method there is no formation of acetylmethylcarbinol in the sponge. The formation starts immediately in the dough when the sugar is added and the A.F. is very close to its maximum by the time the dough is ripe.

### 6. *Effect of Hydrogen Acceptors or Oxidizing Agents and Oxygen*

According to our fermentation scheme as explained before, the presence of hydrogen acceptors in a dough will tend to increase the amount of acetylmethylcarbinol formed by the yeast. Also, the presence of these hydrogen acceptors will tend to prevent reduction of acetylmethylcarbinol formed to 2–3 butylene glycol. Hydrogen acceptors may be oxidizing agents such as potassium bromate, hydro-

gen peroxide, etc., or oxygen. An example of the action of oxygen is given in the following test.

Two sponges were prepared, each consisting of 550 g. flour, 20 g. yeast, 5 g. malt extract, and 340 g. water. One sponge was mixed for 5 minutes in a special dough mixer and oxygen was led through during this time. This sponge was fermented till ripe and mixed into a dough with 450 g. flour, 40 g. sugar, 20 g. salt, 50 g. condensed milk, 20 g. shortening, 205 g. water. This mixing was done in the same dough mixer for 8 minutes in an oxygen atmosphere. The dough was fermented at 80° F. and proofed at 90° F.

The second sponge was treated in exactly the same way and made up into a dough except that no oxygen was led through the sponge and the mixing of the dough was done in the air without special oxygen addition. Samples were taken at regular intervals to determine the A.F. of sponges and doughs. The results are shown in Figure 6.

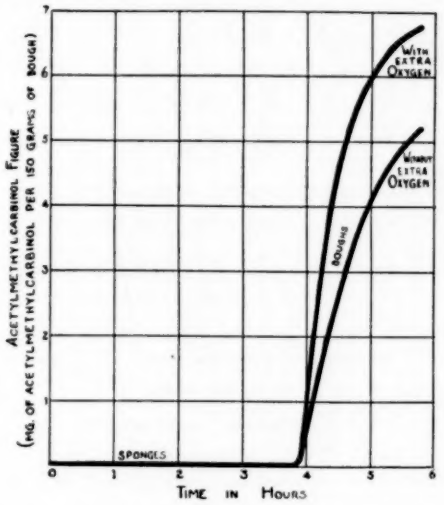



Fig. 6. Influence of oxygen.

In both the oxygen-treated and normal sponges there was no appreciable formation of acetylmethylcarbinol in the sponge, but formation started immediately in the dough after the sugar was added. In the case of the oxygen-treated dough the formation of acetylmethylcarbinol was more rapid and when ready for the oven the A.F. was higher than in the normal dough. Considering the fact that due to practical difficulties, the oxygen-treated dough could only be subjected in a rather imperfect manner for 8 minutes to the influence of oxygen, the results of our tests show the truly remarkable effect of small quantities of oxygen on the formation of acetylmethylcarbinol.

Another example of the influence of oxidizing agents is given in the following test where we studied the effect of additions of potassium bromate in the form of Arkady. We used the sponge dough formula as given in the previous test. One test was run with 3.75 g. Arkady added to the sponge, the second one was run without Arkady. The sponges and doughs were handled in the usual manner. Due to the presence of Arkady, the Arkady sponge was ripe 46 minutes before the control. The results of our determinations are shown in Figure 7.

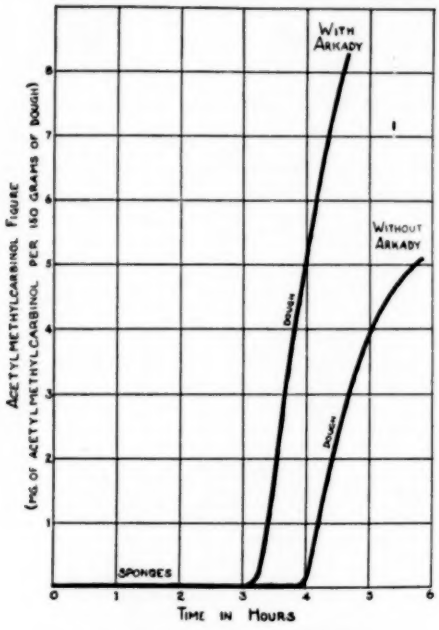



Fig. 7. Influence of oxidizing agents.

We find again that no acetylmethylcarbinol is formed in the sponge. In the presence of oxidizing agents the formation of acetylmethylcarbinol is speeded up considerably in the dough. The final A.F. in the ripe dough is also considerably higher when oxidizing agents are present.

Similar results were obtained in a series of other tests with hydrogen peroxide.

### 7. *Influence of Proper Yeast Selection*

Ordinary samples of commercial yeast contain no acetylmethylcarbinol. All our previously discussed comparative tests were made with the same brand of yeast and every series of tests was made with the same sample of yeast. To study differences in yeast, a series of tests was made using a regular commercial sponge-dough formula in

which the brand and sample of yeast were varied. For these tests the doughs were baked into bread and the A.F.'s were determined on the finished loaves. Table II records the results of these tests.

The values given in Table II are averages of two determinations on the same sample of yeast. Duplicates checked within 0.5 mg.

These results indicate that the type of yeast may have an important influence on the flavor of the bread made with it and therefore this A.F. method may be useful in selecting the proper type of yeast.

It may be well to explain at this point that the A.F. of a ripe dough just before it goes to the oven is a fairly good measure for the A.F. of the finished loaf. During the baking, the yeast activity ceases quickly and the change in A.F. is very small. For comparative tests with various baking formulas and procedures it is not always necessary to follow the A.F. in the dough. It is usually sufficient to determine the A.F. of the finished loaf of bread; *e.g.*, ripe doughs which had A.F.'s of 5.5 and 10.8 gave finished breads with A.F.'s of 5.1 and 11.5. Taking into consideration the loss of weight during baking and the fact that most of this loss is in the crust of the bread, for our purpose these figures check close enough.

TABLE II

INFLUENCE OF COMMERCIAL YEAST ON THE DEVELOPMENT OF A.F

| Commercial yeasts | | |
|---|---|---|
| Brand | Sample | A.F. of bread |
| *A* | 1 | 4.5 |
| | 2 | 6.8 |
| | 3 | 8.1 |
| *B* | 1 | 8.3 |
| | 2 | 11.5 |
| | 3 | 8.3 |
| *C* | 1 | 7.4 |
| | 2 | 7.7 |
| *D* | 1 | 4.8 |
| *E* | 1 | 6.6 |

From the above it will now be clear why, in our first tests, we could not find any acetylmethylcarbinol in breads baked by our regular laboratory method with a formula which was low in sugar and high in yeast. By changing our formula and by using the sponge-dough method we succeeded in baking breads in the laboratory with an A.F. of 11.5, closely approaching the maximum of 15.0 which we found in some commercial loaves.

## Flavor of Bread

From the evidence presented we conclude that acetymethylcarbinol is a natural constituent of bread and is formed by the yeast from the sugar during the fermentation of the dough. This formation is controlled by several factors. Acetylmethylcarbinol present in the ripe dough remains in the bread and there acts as a supply of minute quantities of diacetyl. The rate of oxidation of acetylmethylcarbinol being very slow and the rate of evaporation of diacetyl being very fast, no appreciable accumulations of diacetyl ever occur.

Acetylmethylcarbinol is likely not the only ingredient which controls the flavor of bread. However, it is an important ingredient of bread which leads to the formation of traces of diacetyl and perhaps certain esters which together constitute the essence of the true bread flavor. The presence of acetylmethylcarbinol in sufficient quantity indicates that the bread is produced in a manner and with a formula which enhances the flavor of the loaf. Therefore, further study of acetylmethylcarbinol in its relation to bread flavor should yield further scientific information which will enable the cereal chemist and the baker to produce the ideal loaf of bread with a most appealing flavor.

The determination of acetylmethylcarbinol will never take the place of organoleptic scoring tests but will be an added help in the scoring of bread for flavor in the scientific development of baking formulas and procedures.

## Summary

Acetylmethylcarbinol ($CH_3-CO-CHOH-CH_3$), a substance heretofore never identified with bread was found to be present in most samples of yeast-leavened commercial bread. Acetylmethylcarbinol is formed in the dough during fermentation, as a by-product of the fermentation of the sugar by the yeast. A scheme is presented explaining the formation in accordance with the newer fermentation theories. In the finished loaf of bread acetylmethylcarbinol slowly oxidizes to diacetyl ($CH_3-CO-CO-CH_3$). This last substance which previous investigators found to be responsible for the flavor of butter, is probably also an important constituent of the natural flavor of bread. Details of the method for the determination of acetylmethylcarbinol in dough and bread are given. The term "acetylmethylcarbinol figure" or "A.F." is used to indicate the number of milligrams of acetylmethylcarbinol found in 150 g. of bread dough or in 150 g. of bread crumb. Several figures are given to show the change in A.F. during the fermentation in sponge and straight doughs, with various ingredients and under various conditions.

The following factors have a favorable influence on the A.F.:

High sugar-yeast ratio, diastase preparations, low fermentation temperature, short fermentation time, sponge-dough method, hydrogen acceptors or oxidizing agents and oxygen, proper yeast selection.

**Acknowledgment**

The authors wish to acknowledge the cooperation of Mr. J. J. Hossenlopp who made most of the baking tests.

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# RAPID DETERMINATION OF MOISTURE IN GRAIN [1]

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(Received for publication November 16, 1934)

## Introduction

This paper is a summary of three articles [5] published recently in the Canadian Journal of Research, describing an investigation of the utility and accuracy of certain rapid methods for determining the moisture content of cereals. These included two rapid analytical methods, the Brown-Duvel and 130° C. air-oven, and five electrical moisture meters, the Burton-Pitt, Davies, Limbrick, hand-operated Tag-Heppenstall, and motor-operated Tag-Heppenstall. The vacuum-oven method was employed as a reference standard.

## Experimental Material

The procedure in comparing the general reliability of these methods consisted of determining the moisture content of a subsample of each of some 300 samples of hard red spring wheat, 50 of amber durum wheat, and 75 samples each of oats and barley, by all methods simultaneously, with provision for statistical estimation of sampling errors, errors of method, and ordinary "experimental error." Actually the statistical computations were made on a somewhat smaller number of samples in each case, for in an investigation of this size there were a few inevitable losses due to accidents, etc. Samples of each grain were selected so that about one-third of the total number fell into each of the moisture classes designated as "straight," "tough," and "damp," of which the exact moisture limits are shown in Table I. The grades encountered and the number of samples in each grade were determined by the quality of the grain received at the inspection points during the collection period. In general, and especially with hard red spring wheat, the higher grades were more abundant than the lower ones, but the

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[5] Cook, W. H., Hopkins, J. W., and Geddes, W. F. (1934) Rapid determination of moisture in grain. I. Comparison of 130° C. air-oven and Brown-Duvel methods with vacuum-oven method. Can. J. Research 11: 264–289. II. Calibration and comparison of electrical moisture meters with vaccum oven for hard red spring wheat. *Ibid.*, 409–447. III. Calibration and comparison of electrical moisture meters with vacuum oven for amber durum wheat, barley, and oats. *Ibid.*, 547–563.

samples obtained cover the entire range of moisture content and grade normally encountered in commercial practice.

TABLE I

OFFICIAL MOISTURE LIMITS OF STRAIGHT, TOUGH, AND DAMP GRAIN [1]

| Kind of grain | Percentage moisture (by official Brown-Duvel Method) allowed | | |
|---|---|---|---|
| | Straight Grade | Tough | Damp |
| | % | % | % |
| Wheat | Up to 14.5 | Over 14.5 but not over 17.0 | Over 17.0 |
| Durum wheat | " " 14.8 | " 14.8 " " " 17.0 | " 17.0 |
| Oats | " " 14.0 | " 14.0 " " " 17.0 | " 17.0 |
| Barley | " " 14.8 | " 14.8 " " " 17.0 | " 17.0 |
| Rye | " " 14.0 | " 14.0 " " " 17.0 | " 17.0 |
| Flax | " " 10.5 | " 10.5 " " " 13.5 | " 13.5 |

[1] As established by the Board of Grain Commissioners for Canada.

In addition to the small samples described above, day-to-day variations in the results by the methods under investigation were studied by subdividing three large samples, one in each of the straight, tough, and damp classes, into a large number of subsamples, and determining the moisture content of two subsamples of each daily by the vacuum-oven method, and that of one of each daily by each of the other methods.

One-gallon samples were collected at various points in Western Canada by the staff of the Western Grain Inspection Division. These were shipped in moisture-tight cans to the Dominion Grain Research Laboratory, Winnipeg, where they were cleaned and their approximate moisture content determined. A larger number of samples than required was collected in order to permit selection of the requisite number within each moisture class. Only some 30 samples of naturally damp wheat were received, so that over 70 samples had to be artificially dampened by adding water in order to obtain the necessary damp samples. After dampening with distilled water the samples thus treated were allowed to stand for one month in moisture-tight cans in a room maintained at a temperature near the freezing point. The samples were removed from the cans every few days, thoroughly mixed and replaced in the cans. The naturally straight, tough, and damp samples were also stored under the above conditions for at least a month with occasional mixing before they were subdivided for the various tests. All of the damp samples of oats and barley tested were " naturally " damp when received, but as no naturally damp samples of amber durum wheat were available, certain straight grade and tough samples were moistened by exposure to high humidity for several days, dried back in the laboratory to the required moisture and stored before testing.

A one-to-eight progressive riffle was designed, constructed, and employed for dividing the one-gallon samples into the eight subsamples required, the sampling being done in a room at a temperature near the freezing point. As the samples had all been kept for a month or more in moisture-tight cans with periodic mixing, it was assumed that each sample was reasonably homogeneous with respect to moisture. The subdivision of these relatively small samples into eight subsamples appeared therefore to present a relatively simple sampling problem, and the apparatus described above, after adjustment to give eight subsamples of practically equal size, was considered adequate for this purpose. Nevertheless, in order to obviate any possible systematic error, the eight spouts of the sampler were numbered and the corresponding subsamples distributed at random among the methods and instruments under test. The samples were divided two or three days before actual use and the resulting subsamples were then allowed to attain temperature equilibrium before testing by letting them stand for twenty-four hours in the room in which the determinations were made.

## Vacuum-Oven Method

### Procedure

As some of the samples to be tested were of quite high moisture content, it was thought that a significant loss of moisture might occur during grinding and the temporary exposure of the samples to the atmospheric conditions of the room in weighing, etc. Preliminary experiments substantiated this assumption and a two-stage drying procedure was therefore employed. The first stage consisted of drying $100.00 \pm 0.01$ g. of grain to equilibrium with an atmosphere of 40% relative humidity at 72° F., re-weighing and grinding under the same atmospheric conditions in a Wiley mill fitted with a 1 mm. sieve. The second stage of the drying was carried out by the official vacuum-oven method, using a 16-hour heating period, as it was found that constant weight over an additional 2-hour heating period was not attained in a shorter time when the oven was filled with drying dishes.

Two of the subsamples, obtained from the subdivision of each sample, were employed for the vacuum-oven determination. Comparison of the results obtained from the two subsamples made possible an estimate of the sampling error. As two DeKhotinsky ovens were required to accommodate the number of samples tested daily, the difference between ovens was studied by placing one of the duplicates of each subsample in each of the two ovens. The ovens employed were identical in construction, and were operated under the same conditions of temperature and pressure.

## Results by Vacuum-Oven Method

The individual results obtained from the hard red spring wheat samples were subjected to an analysis of variance, the total variance being divided into portions attributable to average difference between ovens, differences between subsamples and the difference between duplicates after eliminating any average difference between ovens. The need for determining the sampling error in an investigation of this type was demonstrated by the fact that the variance between subsamples was significantly greater than that between duplicates of the same subsample, even in this relatively simple sampling process, which merely consisted of subdividing a small sample. There was also a significant difference between ovens which demonstrates that ovens of identical construction, operated under the same conditions, may give slightly different results. The results of the foregoing analysis, expressed in terms of percentage of moisture, are summarized in Table II together with statistics indicating the degree of accuracy attained with the other grains, amber durum wheat, oats, and barley.

TABLE II

Results of Analysis of Variance of Vacuum-Oven Determinations

| Hard Red Spring Wheat | |
|---|---|
| | *Moisture* % |
| Average difference between ovens | 0.071 |
| Standard error of duplicates ("experimental error") [1] | 0.044 |
| Standard error of sampling | 0.110 |
| Standard error of duplicates plus sampling error [1] | 0.118 |
| Day-to-day variations (standard deviation) | 0.084 |
| **Other Grains** | |
| Standard error of duplicates plus sampling error [1] | |
| Amber durum wheat | 0.093 |
| Barley | 0.089 |
| Oats | 0.109 |

[1] Standard errors apply to mean of duplicates.

The results obtained from the eleven daily analyses of two subsamples of the three large samples of hard red spring wheat previously mentioned, showed a correlation between fluctuations in apparent moisture content of all three samples from day to day. This finding was confirmed by an analysis of variance which showed that the variance due to daily fluctuation, common to all three samples, was significantly greater than the residual variance attributable to sampling errors, etc. The net variance attributable to daily variation corresponded to a standard error of 0.084% moisture. It is probable that the average differences between ovens and the day-to-day variation are to some extent

different measurements of the same effect. Owing to the smaller number of samples of the other grains tested, the experiments with any one were usually completed within a two or three-day period, and with these the effect of daily variations in the vacuum oven is less evident, but the number of days available is too small to yield a reasonably reliable estimate of the variance to be expected.

## Rapid Analytical Methods

### Procedure

Although the grain must also be ground for the air-oven method, it is obviously out of the question to use the two-stage drying procedure employed with the vacuum-oven method. Furthermore, although the 130° C. air oven is frequently employed for determining the moisture content of grain, it is only recognized as an official method for flour. It appears therefore that the results by this method may depend somewhat on the method and extent of grinding. Two types of grinder are commonly used, the attrition type and the shearing type, and these were represented in the present investigation by the Hobart grinder and Wiley mill respectively. The main series of 300 hard red spring wheat samples was divided into two lots of 150, one lot being ground in the Hobart and the other in the Wiley mill. Owing to the superiority of the latter all of the other grains were ground in it. Otherwise the 130° C. air-oven method, official for flour, was employed.

Since it was felt that the 130° C. air-oven would probably be the most accurate of the rapid methods, the portions required for the former method were taken from one of the subsamples employed for the vacuum-oven determinations in order to avoid a sampling error possibly large enough to vitiate an estimate of its accuracy in comparison with the vacuum oven.

The Brown-Duvel tester was operated in accordance with the official methods, but with the additional refinements introduced by the Dominion Grain Research Laboratory. These, in addition to careful calibration of all flasks, cylinders and thermometers, include specially made electrical heaters, careful regulation of line voltage, and close control of room temperature and of the time required for heating and cooling the flasks.

### Results by 130° C. Air-Oven Method

It was found that the air-oven method, regardless of the method of grinding employed, consistently underestimated the amount of water present, the extent of this underestimation increasing with the actual moisture content. It is probable that this underestimation, as compared

with the vacuum oven, is due to a loss of moisture during grinding and subsequent manipulation, since the grain was ground directly without preliminary drying. This systematic bias was eliminated by calculating, by the method of Least Squares, a linear regression equation expressing the expected vacuum-oven moisture in terms of the observed air-oven result, for both methods of grinding. These equations, together with the standard error of duplicates, and the net standard error of prediction of the vacuum-oven values after applying the correction equation and allowing for the variance due to the experimental error in the vacuum-oven determination, are shown in Table III.

TABLE III

STANDARD ERROR OF DUPLICATES, CORRECTION EQUATIONS AND NET STANDARD ERROR OF PREDICTION OF VACUUM-OVEN PERCENTAGE MOISTURE OF GRAIN BY THE 130° C. AIR-OVEN AND BROWN-DUVEL METHODS

| Determination | Standard error of duplicates, moisture [1] | Correction equation [2] | Net standard error of prediction, moisture [1] |
|---|---|---|---|
| | % | *130° C. air oven* | % |
| Hard red spring wheat (Hobart) | 0.069 | $M_v = -0.644 + 1.066M_a$ | 0.30 |
| Hard red spring wheat (Wiley) | 0.050 | $M_v = -0.926 + 1.095M_a$ | 0.24 |
| Amber durum wheat | 0.052 | $M_v = -0.452 + 1.084M_a$ | 0.12 |
| Barley | 0.056 | $M_v = -1.548 + 1.131M_a$ | 0.20 |
| Oats | 0.057 | $M_v = -2.225 + 1.195M_a$ | 0.20 |
| | | *Brown-Duvel* | |
| Wheat | 0.053 | $M_v = -0.280 + 1.042M_{BD}$ | 0.16 |
| Amber durum wheat | 0.040 | $M_v = +1.026M_{BD}$ | 0.09 |
| Barley | 0.034 | $M_v = -0.046 + 1.005M_{BD}$ | 0.12 |
| Oats | 0.042 | $M_v = -0.674 + 0.983M_{BD}$ | 0.13 |

[1] Standard errors apply to mean of duplicates.
[2] $M_v$ = moisture content as determined by vacuum oven.
$M_a$ = moisture content as determined by 130° C. air oven.
$M_{BD}$ = moisture content as determined by Brown-Duvel.

It is evident from this table that the standard error of the duplicates, commonly taken as a measure of the "experimental error," although somewhat larger for the air oven than for the vacuum oven, is in all cases small compared with the net error of prediction of moisture content. This indicates that although duplicates may check fairly well within themselves, there may be a relatively large discrepancy in predicting the moisture content by the vacuum-oven method from results obtained in the air oven. The results in Table III also show that the standard error of prediction, and of the duplicates, are lower with the Wiley than with the Hobart grinder, showing the former mill to be superior for this determination. The standard error of prediction with

amber durum wheat, barley, and oats, using the Wiley mill, is in all cases somewhat lower than that obtained with hard red spring wheat, while the standard errors of the duplicates are about the same.

Upon analysis, the results, obtained from the three samples tested daily by the air-oven method for the purpose of studying daily variation, did not reveal consistent daily fluctuations. Nevertheless, in view of the known existence of daily variation in the vacuum-oven results, the effect of eliminating daily differences from the comparisons of the two methods in the general reliability tests was determined by computing the variances and covariance about the daily means instead of about the mean of the entire series. The resulting reduction in the standard errors of prediction indicated that the daily fluctuations in the air-oven method were small when the Wiley mill was employed, but were significant when the Hobart grinder was used, doubtless due to the difficulty of maintaining an identical setting of the machine from day to day.

### Results by Brown-Duvel Method

The results showed similar characteristics to those obtained with the air oven, namely an underestimation of the moisture content which, with hard red spring and amber durum wheat, increases as the actual moisture increases. The underestimation can be explained by the fact that the Brown-Duvel was originally developed to give results comparable with the old water-jacketed drying-oven method, which is known to give lower values than the vacuum oven. The progressively greater underestimation of the moisture content of wheat, as the actual moisture increases, must be attributed to the conditions of the test, probably to a variation in the heating time with grain of different moisture content, since it is known that slow heating gives low results and *vice versa*.

The systematic bias was eliminated, as before, by computing a linear regression equation appropriate to the results obtained with each of the four grains. These equations, together with the standard errors of duplicates, and the net standard error of prediction of the observed vacuum-oven values after allowing for the sampling error and experimental error of the vacuum-oven method are given in Table III. The standard error of the duplicates is again quite small compared with the standard error of prediction, and of the same order, or slightly smaller, than that obtained with the air oven. The standard error of prediction, however, is in all cases significantly lower for the Brown-Duvel. Since this proved to be the most accurate rapid analytical method, the results obtained with the Brown-Duvel for hard red spring wheat are shown plotted against the corresponding vacuum-oven results in Figure 1. The underestimation of the vacuum-oven percentage moisture is evident

from the figure which is typical of the results obtained with both the Brown-Duvel and air-oven methods with all grains.

The three samples tested daily by the Brown-Duvel method showed no evidence of daily fluctuations under the conditions of this investiga-

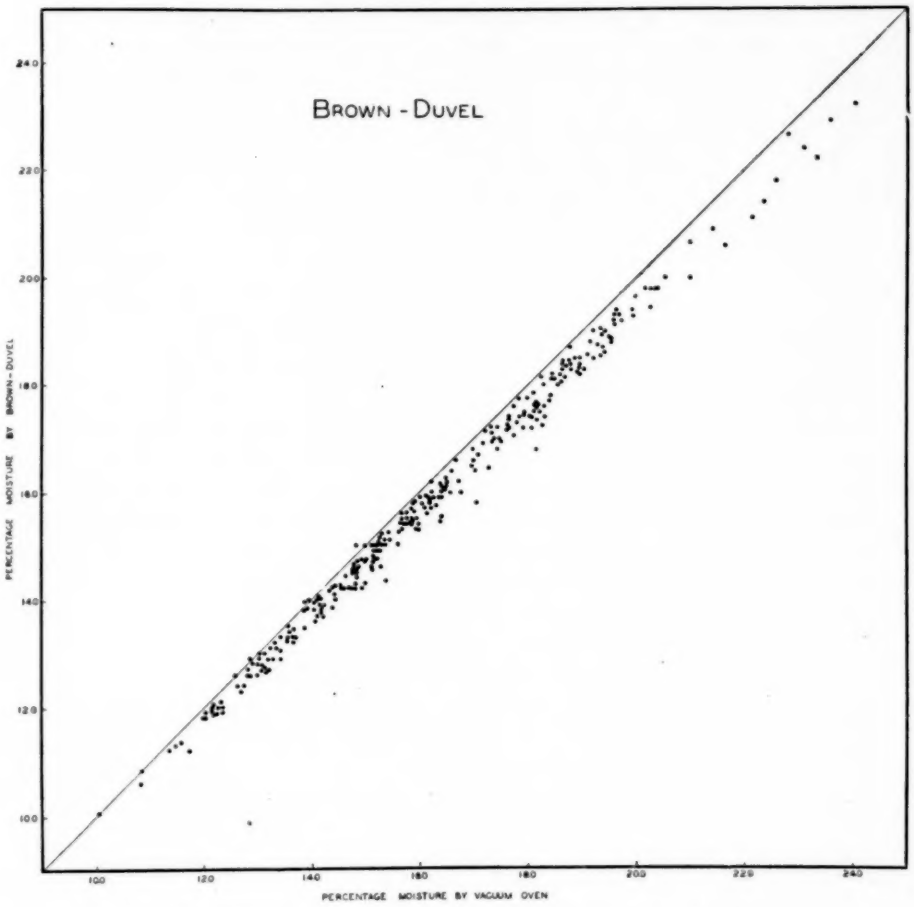



**Fig. 1. Moisture content of hard red spring wheat samples by Brown-Duvel and vacuum-oven methods.**

tion. Calculation of the variances and covariance of the general reliability series results around the daily means instead of around the general mean, however, indicated that day-to-day variations had been a definite source of error in comparing this method with the vacuum oven, but it seems probable that this can be attributed entirely to daily fluctuations, already demonstrated, in the vacuum-oven results.

## Electrical Moisture Meters

### Practical Features of Meters Studied

Electrical moisture meters for grain may be designated as "dielectric" or "resistance" types depending on the principle employed. The "dielectric" type was represented in this investigation by the Burton-Pitt, all the others studied being of the resistance type. A number of the practical features of the instruments, including accessory equipment, time, and space for operation required, are given in Table IV. The Tag-Heppenstall instruments are the quickest in operation, and require no accessory equipment. The grain must be weighed for the Burton-Pitt and ground for the Limbrick, a special hand-operated burr grinder being supplied with the instrument.

TABLE IV

Practical Features of Electrical Moisture Meters Studied

| Meter | Accessory equipment required | Power required | Type of test cell | Operating space required | Average time required for duplicate tests[1] | Remarks |
|---|---|---|---|---|---|---|
| | | | | *Sq. ft.* | *Minutes* | |
| Burton-Pitt | Balance for weighing sample to ± 0.01 g. | Power for rectifier to charge wet cell | Condenser | 6 | 2.4 | Instrument must be allowed to warm up 20 to 30 minutes before use |
| Davies | None. Grain must be packed in test cell | Self-contained | Electrodes in bottom and wall of cell | 2 | 3.7 | |
| Limbrick | Grinder, supplied with meter | Self-contained | Electrodes in wall and centre of cell | 4 | 5.4 (3.6) | Time for triplicate tests as recommended by maker. Figure in brackets for duplicate tests |
| Tag-Heppenstall (hand) | None | Self-contained | Small roller electrodes | 4 | 1.5 | |
| Tag-Heppenstall (motor) | None | Power required to operate fractional h.p. motor | Large roller electrodes | 5 | 1.2 | |

[1] Average time required for 120 duplicate tests, including weighing or grinding if necessary, but not time for selecting samples, reading conversion charts or making interpolations.

### Procedure in Operating Electrical Moisture Meters

All the instruments were operated in accordance with the instructions supplied by the manufacturers, and no particular effort was made to determine the best technique, operating conditions or settings. The

meters were tested in a room conditioned as to temperatrue and humidity, but owing to the number of men working in the room, the temperature sometimes rose 2 or 3° F. above the desired value, and it was necessary to correct the results to a constant temperature basis. Two operators read the five meters, and in order to avoid any systematic error due to personal factors, a separate random allotment of meters and operators was made for each sample. The temperature of the grain was determined just before testing.

### Results Obtained with Electrical Moisture Meters

The Davies meter was found to be of little value for determining the moisture content of hard red spring wheat. It was not sensitive enough to give appreciable deflections with samples containing less than 16.0% moisture, and gave extremely erratic results at higher moisture contents. In consequence, no attempt was made to calibrate this meter and it was excluded from further experiments.

The Burton-Pitt meter was calibrated against the vacuum oven over the moisture range 11.0 to 17.0%, with hard red spring wheat, the calibration curve being fitted to the observations by the method of Least Squares. At higher moisture contents this instrument gave exceedingly erratic results. With the other grains the range over which the meter was calibrated was determined by the quality of the results, and with oats and barley it was even shorter than with wheat. The calibration curves were all slightly parabolic. Their equations, together with the experimental error, and the net standard error of prediction of moisture, after allowing for the variance due to sampling error and experimental error in the vacuum-oven method are given in Table V. The experimental error is again in all cases small compared with the error of prediction. The meter appears to be least accurate with barley and more accurate with oats than with the wheats, but it must be remembered that it was calibrated only over a limited arbitrarily chosen moisture range for the two coarse grains. However, the errors of prediction are all so high that the meter is judged as being of little practical utility.

The Limbrick and the two Tag-Heppenstall meters operate on the resistance principle and it was found that all of these meters exhibited a linear relationship between moisture content and the logarithm of the resistance up to about 17.0% moisture. At higher moisture contents the logarithmic resistance—moisture relation becomes parabolic; successive increments of moisture causing progressively smaller diminutions in resistance, and the estimation of the moisture content therefore becoming less precise. In view of the character of the resistance moisture relationship, separate curves were fitted, as before, to the results

with each instrument for each grain tested, in the range below and above 17.0% moisture. The errors appropriate to each moisture range were also computed separately. The experimental errors, calibration equations, and net standard errors of prediction are given in Table V.

It is evident that the meters are in nearly all cases more accurate

TABLE V

EXPERIMENTAL ERROR, CALIBRATION EQUATIONS, AND NET STANDARD ERROR OF PREDICTION OF VACUUM-OVEN MOISTURE CONTENT OF GRAIN BY ELECTRICAL MOISTURE TESTERS

| Determination | Moisture range | Experimental error (standard error of duplicates), moisture[1] | Calibration equation[2] | Net standard error of prediction, moisture[1] |
|---|---|---|---|---|
| | % | % | | % |
| | | | *Burton-Pitt* | |
| Hard red spring wheat | 11.0–17.0 | 0.06 | $M_v = 6.87 + 0.279x - 0.00152x^2$ | 0.54 |
| Amber durum wheat | 11.5–18.5 | 0.06 | $M_v = 7.56 + 0.199x - 0.000718x^2$ | 0.44 |
| Barley | 11.0–16.0 | 0.09 | $M_v = 4.42 + 0.401x - 0.00340x^2$ | 0.83 |
| Oats | 12.5–15.5 | 0.05 | $M_v = 8.23 + 0.244x - 0.00186x^2$ | 0.39 |
| | | | *Limbrick* | |
| Hard red spring wheat | 11.5–17.0 | 0.08 | $M_v = 18.39 - 1.96 \log R$ | 0.38 |
| | 17.0–21.5 | 0.09 | $M_v = 19.43 - 4.65 \log R + 2.32 (\log R)^2$ | 0.49 |
| Amber durum wheat | 11.5–16.0 | 0.04 | $M_v = 17.07 - 1.79 \log R$ | 0.29 |
| | 16.0–19.0 | 0.05 | $M_v = 17.84 - 3.52 \log R + 1.16 (\log R)^2$ | 0.30 |
| Barley | 11.0–17.5 | 0.07 | $M_v = 18.61 - 2.44 \log R$ | 0.42 |
| | 17.5–20.0 | 0.06 | $M_v = 19.65 - 6.62 \log R + 4.28 (\log R)^2$ | 0.50 |
| Oats | 12.5–17.0 | 0.12 | $M_v = 18.78 - 2.38 \log R$ | 0.30 |
| | 17.0–22.0 | 0.19 | $M_v = 20.74 - 8.88 \log R + 6.56 (\log R)^2$ | 0.57 |
| | | | *Tag-Heppenstall (hand)* | |
| Hard red spring wheat | 11.0–17.5 | 0.04 | $M_v = 23.78 - 2.46 \log R$ | 0.40 |
| | 17.5–24.0 | 0.05 | $M_v = 28.77 - 7.39 \log R + 1.15 (\log R)^2$ | 0.44 |
| | | | *Tag-Heppenstall (motor)* | |
| Hard red spring wheat | 11.0–17.0 | 0.02 | $M_v = 21.93 - 2.48 \log R$ | 0.16 |
| | 17.0–24.0 | 0.03 | $M_v = 26.21 - 8.08 \log R + 1.82 (\log R)^2$ | 0.56 |
| Amber durum wheat | 11.5–17.5 | 0.02 | $M_v = 21.00 - 2.38 \log R$ | 0.13 |
| | 17.5–22.0 | 0.04 | $M_v = 26.05 - 8.81 \log R + 1.99 (\log R)^2$ | 0.10 |
| Barley | 11.0–17.5 | 0.04 | $M_v = 21.96 - 2.75 \log R$ | 0.29 |
| | 17.5–22.0 | 0.06 | $M_v = 28.66 - 10.38 \log R + 2.24 (\log R)^2$ | 0.34 |
| Oats | 12.5–17.5 | 0.07 | $M_v = 23.90 - 2.85 \log R$ | 0.55 |
| | 17.5–22.5 | 0.13 | $M_v = 32.58 - 9.84 \log R + 1.55 (\log R)^2$ | 1.00 |

[1] Standard errors apply to mean of duplicates.
[2] $M_v$ = moisture content as determined by vacuum oven.
$x$ = meter scale reading at sensitivity of 90.
$R$ = resistance in megohms for Limbrick, and in 1000's of ohms for Tag-Heppenstall meters.

in the lower than in the higher moisture range. In general, these three meters are all more accurate than the Burton-Pitt or Davies. From 11.0 to 17.0% moisture, which is the most important range commercially, the motor-operated Tag-Heppenstall is superior to the Limbrick with all grains except oats, the latter instrument being judged second in merit. The hand-operated Tag-Heppenstall, although somewhat more accurate than the Limbrick with hard red spring wheat, was found to be unsatisfactory with the other grains, as they would not

feed into the roller electrodes in a satisfactory manner. The Limbrick and both Tag-Heppenstall meters were more accurate with the two types of wheat than with the two coarse grains. In all cases the experimental error is again small compared with the error of prediction. This indicates that the meters are capable of measuring the resistance with sufficient accuracy, but that the resistance itself is determined to some extent by some physical property other than moisture content. This property may be inherent in the grain or it may be induced by the instrument, *e.g.*, polarization.

The results obtained with hard red spring wheat in the motor-operated Tag-Heppenstall instrument are shown in Figure 2, and are typical of the results obtained with the three better resistance type meters with all grains. Three curves were fitted in the damp moisture range, representing respectively naturally damp, artificially dampened, and all samples (broken line). The two curves for the artificially and naturally damp wheat proved to be significantly different but on further analysis it seems likely that part, if not all, of the observed difference can be attributed to daily fluctuations in the vacuum-oven results rather than to the method of wetting. The results obtained with all meters were naturally affected by the daily variations evident in the vacuum-oven determinations, but an analysis of the three large samples tested daily by all meters failed to give definite evidence of consistent daily fluctuations, except with the Burton-Pitt where there was some evidence to indicate that the meter results were also affected.

Comparison of the results from a limited number of Garnet samples with those obtained in the general reliability series from typical commercial hard red spring wheats, indicated that for a given meter, the same calibration curve was applicable, with the exception of the hand Tag-Heppenstall where the difference can probably be attributed to an unsuitable roll spacing.

The range of moisture covered by the meters is not identical. The upper limit is in all cases determined by the increasing uncertainty of the results. The lower limit of the Limbrick is about 11.5%, while the hand Tag-Heppenstall can be used down to 9.0% moisture, and the motor model down to 7.0% moisture. The calibration equations given in Table V can be used to cover this range with reasonable accuracy. The Burton-Pitt gave appreciable deflections even with the driest wheat tested, but the calibration equation obtained at higher moisture contents cannot be used in this range.

The temperature coefficients of both wheat alone and the wheat-instrument system were determined and found to differ appreciably in the Burton-Pitt and Limbrick testers. Both coefficients were found to be dependent on the moisture content of the grain. The coefficients,

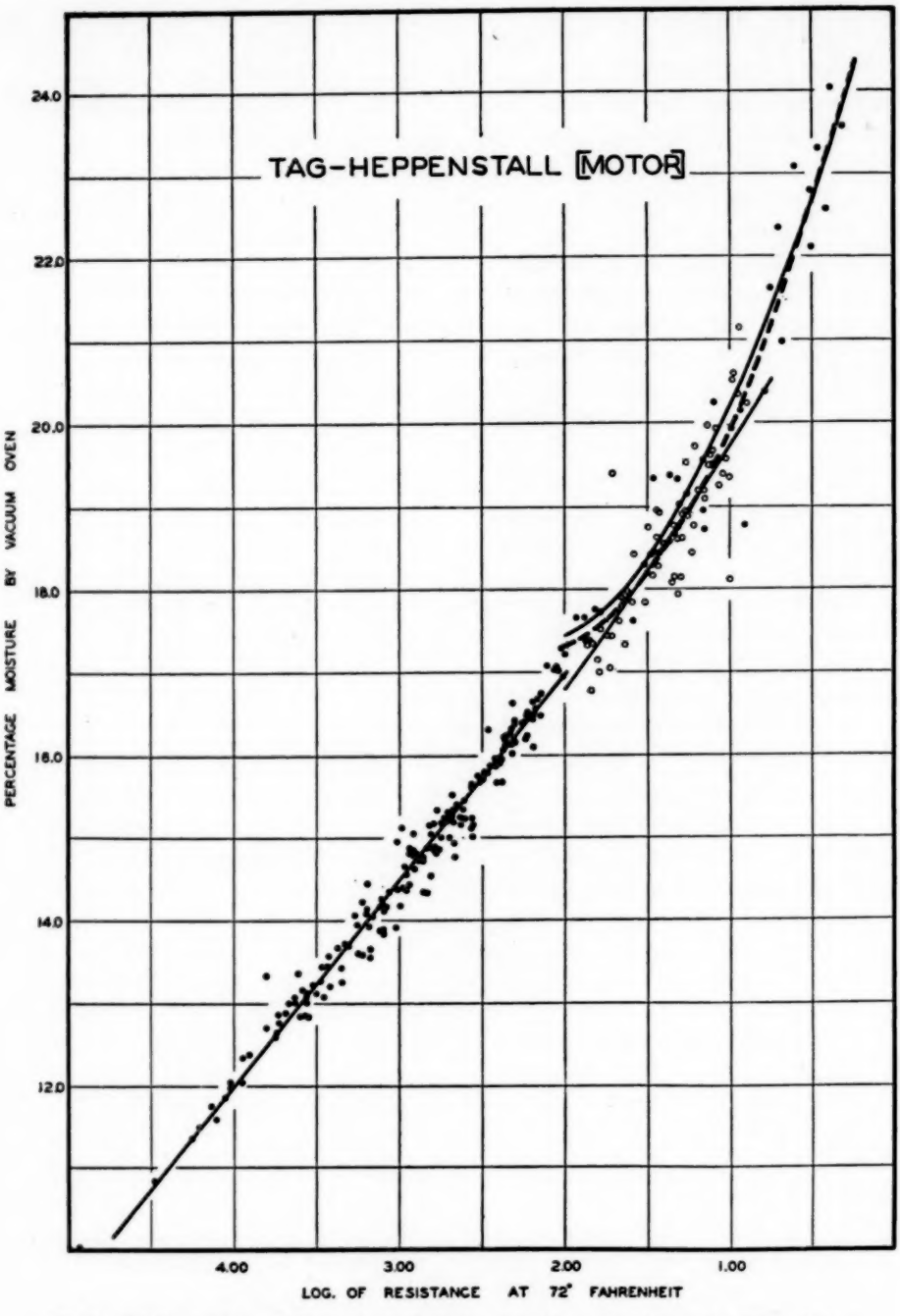



Fig. 2. Relation between moisture content of wheat and logarithm of resistance in 1,000's of ohms. Motor-operated Tag-Heppenstall meter. Hollow circles represent artificially dampened samples.

in terms of resistance or meter scale reading, were converted to the equivalent moisture content using the appropriate calibration equation, and are shown in terms of corrections per ° F. for the various meters in the different moisture ranges in Table VI. The temperature coef-

TABLE VI

TEMPERATURE CORRECTIONS IN TERMS OF PERCENT. MOISTURE

Corrections are to be *added* if the temperature is below the standard, and *subtracted* if above the standard.

| Meter | Standard temperature | Correction in terms of percent. moisture for each ° F. above or below the standard temperature | | | | | |
|---|---|---|---|---|---|---|---|
| | | Straight | | Tough | | Damp | |
| | | Wheat and meter | Wheat alone | Wheat and meter | Wheat alone | Wheat and meter | Wheat alone |
| | ° *F.* | % | % | % | % | % | % |
| Burton-Pitt | 72 | 0.071 | 0.041 | 0.081 | 0.047 | — | — |
| Limbrick | 75 | 0.069 | 0.031 | 0.067 | 0.040 | 0.052 | 0.037 |
| Tag-Heppenstall (hand) | 72 | 0.054 | — | 0.057 | — | 0.066 | — |
| Tag-Heppenstall (motor) | 72 | 0.067 | 0.060 | 0.068 | 0.060 | 0.068 | 0.068 |

ficients for the other grains appear to be practically the same as those obtained with hard red spring wheat. Humidity coefficients were also determined but were found in all cases to be small compared with those of temperature, in general a change of 10% in relative humidity at 72° F. having less effect than a change of 1° F. in temperature.

## Conclusions

Comparison of the results obtained by the rapid analytical methods and moisture meters shows that the Brown-Duvel is as accurate with hard red spring wheat throughout the entire moisture range studied, as the motor-operated Tag-Heppenstall is over the limited range 11.0 to 17.0%. At higher moisture contents, or with any of the other grains used the former method is superior in accuracy to any of the electrical testers. The 130° C. air-oven method, using a Wiley mill for grinding, is also more accurate than any of the meters except the motor-operated Tag-Heppenstall in the low moisture range with hard red spring wheat.

The results as a whole indicate that the motor Tag-Heppenstall is the most accurate of the moisture meters examined, for all grains except oats, for which the Limbrick is best. Additional points of superiority

of the motor-operated Tag-Heppenstall include a more extended moisture range, greater rapidity of operation, and a negligible instrumental temperature coefficient as compared with the other instruments. The Limbrick is next in order of merit, although considerably slower in operation.

### Acknowledgments

The authors are indebted to the several individuals and manufacturing companies referred to in the original papers, whose co-operation materially facilitated the progress of the investigation.

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## THE GROWTH OF BREAD MOLDS AS INFLUENCED BY ACIDITY

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### Introduction

It is well-established, commercial practice to add weak acids or acid salts to doughs for the purpose of controlling "rope," a disease of bread caused by bacteria of the Mesentericus type. It is of importance to know what effect such acid constituents have on the development of molds on bread. Within the limits of bread-making, does acidity stimulate or inhibit the growth of molds? Thom and Church (1926), Bakers' Weekly (1929), Richards (1933), Skovholt and Bailey (1933), and Pirrie (1934), have stated that acidity favors or stimulates the growth of molds, but gave no experimental evidence to support their conclusion. In the baking industry, there is much difference of opinion on the subject.

The work of many investigators, Clark (1899), Thom (1910), Webb (1919, 1921), Steinberg (1919), Johnson (1923), Acklin and Schneider (1928), Kadelbach (1931), Marloth (1931), and Iuracec (1932), shows that a slightly acid medium is generally more favorable to the growth or development of molds than is a neutral or slightly alkaline medium. This generalization, however, does not, in itself, justify a deduction that a pH of 5.0, for example, is more favorable to the growth of molds than is a pH of 6.0. In fact Duggar, Severy, and Schmitz (1917) reported that *Aspergillus niger* grew well in a natural mangold decoction at pH 4.5, but produced a heavier mat when the medium was brought to pH 6.0. Furthermore, the experimental evidence presented by Johnson (1923), Acklin and Schneider

(1928), and Marloth (1931), indicates that molds, like yeasts, do not exhibit any well-defined growth optimum with respect to hydrogen-ion concentration, but grow equally well over a rather wide range. From their work, it would seem that this range covers the entire range of hydrogen-ion concentration within which commercial bread is produced.

Studies dealing with the influence of acids on the growth of microorganisms must take into consideration not only the effect of hydrogen-ion concentration but also the specific effect of the particular acids under investigation. In investigating the effect of various acids upon the growth and development of *Aspergillus niger* in Raulin's solution, Kiesel (1913) showed that certain acids exhibit a marked toxicity. Fatty acids, formic and acetic, for example, were found to be more toxic toward the fungus than acids such as hydrochloric, sulfuric, and phosphoric. Clark (1899) found acetic acid more toxic to mold fungi than mineral acids, and attributed its toxicity to the undissociated molecule. Reichel (1910) showed that acetic acid had a marked harmful effect on the development of a *Penicillium*, and demonstrated that it was more toxic to this mold than were mineral acids. He conducted experiments which showed that the toxicity is due to the undissociated acid and not to the acetate ion. Cruess and coworkers (1929, 1931), reported that the concentrations of sodium benzoate, potassium acetate, and sodium sulfite required to prevent the growth of yeasts, molds, and bacteria were much less at pH 2.0 to 4.5 than at pH values of 5.0 to 9.0. They concluded that the undissociated acids, and not the anions, were the toxic agents. Furthermore, their work indicates that hydrogen-ions play, for the most part, an indirect role in the effect of acids on mold growth.

All of the investigations cited were carried out with media differing considerably from bread, and for this reason could not be unhesitatingly applied to practical bread-making problems. Recently Nielsen and Hartelius (1933) determined the effect of hydrogen-ion concentration on the growth of *Aspergillus niger*, using in one series of experiments a purely synthetic medium, and in another the same medium plus a widely occurring substance, which they called a growth-promoting substance. They gave evidence for a very important and previously neglected consideration, namely, that one may be greatly deceived in assuming that the pH optimum for the growth of a fungus is the same in a natural medium as it is in a synthetic medium. The addition of the growth-promoting substance to their synthetic medium, for example, caused a shift in the growth optimum from pH 2.5–3.0 to pH 6–7.

From the foregoing summary of the literature, it is evident that

there is a lack of published experimental data of direct practical value to the baking industry. A study of some of the factors involved in the question of mold growth on bread versus acidity was therefore undertaken. Pure cultures of a number of common molds were obtained for this work. Only one of these, namely, *Aspergillus niger*, has been studied to date. Investigations of other molds will be published later.

## Medium and Method

It seems obvious that the choice of a culture medium is of paramount importance if results are to have any practical significance. It would be preferable to use the bread loaf itself as a substrate. However, a quantitative approach to the problem necessitates the use of a liquid medium from which the mold crops may be harvested and weighed at intervals after inoculation. In addition to this consideration, a liquid medium offers other advantages in general experimental technique. It was therefore decided to use an aqueous extract of bread as the culture medium for the principal studies of this investigation. This medium was found to support a luxuriant growth of all cultures of bread molds available. It will be shown later that a certain degree of correlation prevails between results obtained with the liquid medium and with the bread loaf itself.

The bread used in the preparation of the "bread medium" was made by the following formula:

| Sponge | Dough |
|---|---|
| 15 lbs. flour | 10 lbs. flour |
| 9¾ lbs. water | 5½ lbs. water |
| 1 oz. dough improver | 1¼ lbs. sugar |
| 8 oz. yeast | 12 oz. fat |
| 4 oz. malt extract | 1 lb. powdered skim-milk |
| | 8 oz. salt |

To prepare the medium, 3 one-pound loaves were broken up and mixed with 4 liters of water in a cake mixer. The water was taken at 35° C. The mixture was well beaten and then poured on large fluted papers to filter overnight. The filtrate thus obtained was not quite clear. In order to free it from suspended matter, it was treated with diatomaceous earth, boiled, and filtered while hot on large Buchner funnels. The resulting filtrate had a pH of 4.9–5.3. Analysis of the medium so made, follows:

| | | |
|---|---|---|
| Total solids (100° C. in vacuum) | 2.58 | gms. per 100 cc. |
| Ash (550° C.) | 0.44 | " " " " |
| Protein (N×5.70) | 0.0586 | " " " " |
| Reducing sugar (as invert sugar) | 0.99 | " " " " |
| Total sugar (after inversion) | 1.03 | " " " " |

Sufficient medium for all the experiments described was prepared at one time, distributed in 6 liter flasks, and sterilized in flowing steam. This was the medium used as "bread medium," except where noted.

Buffering the medium is practically impossible, since *Aspergillus niger*, the organism chosen for this paper, produces considerable acid during growth. The addition of phosphate or acetate buffer, sufficient to interfere with growth of the organism, did not hold the pH even approximately constant.

Nielsen and Hartelius (1933), in their work with *Aspergillus niger*, were forced to consider only the initial pH and its effect on growth. They found, however, that the initial pH had considerable effect. Our results entirely corroborated their findings; hence the work described in this paper was confined to the effect of initial pH. This situation might be considered somewhat fortuitous since the bread loaf itself is not a very well buffered substrate. The effect of the pH of bread on the growth of molds will be, for the most part, a function of the initial pH of the bread.

## Experimental Procedure

### Part I

### *Effect of Initial Hydrogen-ion Concentration upon the Growth of Aspergillus niger*

This experiment was performed twice. The first time (Table I) the bread medium was diluted with an equal volume of water. The second time (Table II) the medium was used as prepared, as was the case in all succeeding experiments. This will serve to explain the discrepancy in yields between Table I and Table II.

The initial pH of the medium was adjusted by means of concentrated hydrochloric acid and sodium hydroxide. Very small volumes of these reagents were required; thus any significant change in the concentration of the bread medium was avoided. The quantity of chloride and sodium ions introduced into the medium was far too small to have any detectable effect on the growth of the mold, a fact which was accepted only after checking with an excessive quantity of sodium chloride. All pH measurements were made electrometrically.

50 cc. portions of bread medium, the pH of which had been adjusted to the desired level, were pipetted into sterile 200 cc. Erlenmeyer flasks. These were plugged with cotton and sterilized in flowing steam on two successive days. Each flask was then inoculated with 1 cc. of a suspension of *Aspergillus niger* conidia in pure culture.[1] The total volume in each flask was therefore 51 cc. After inoculation, the flasks were placed in an incubator, where growth took place at 30° C. in darkness.

[1] This suspension of conidia was prepared by filtering a well-shaken, 72-hour culture of *Aspergillus niger* through sterile cotton.

The dry weight of mold, which had developed in different intervals of time, was determined by filtering the contents of the flasks through weighed Alundum crucibles. The crops were thoroughly washed with distilled water, after which the crucibles and contents were dried at 100° C. to constant weight. The crops were recorded in milligrams of dry matter. Crops were harvested in triplicate at each pH level, and pH measurements were made on the combined filtrates of the three crops. The results are summarized in Tables I and II.

TABLE I

GROWTH OF *Aspergillus niger* AS INFLUENCED BY INITIAL PH

| Acidity of medium | Time of growth, *hours* | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | 26 | | 45 | | 69 | | 93 | | 141 | |
| Initial pH | Dry matter | pH of medium | Dry matter | pH of medium | Dry matter | pH of medium | Dry matter | pH of medium | Dry matter | pH of medium |
| | *mgs.* | | *mgs.* | | *mgs.* | | *mgs.* | | *mgs.* | |
| 1.6 | Trace | 1.6 | 10±1 | 1.6 | 33±0 | 1.6 | 56±2 | 1 6 | 67±1 | 1.6 |
| 1.9 | Trace | 1.9 | 13±2 | 1.9 | 40±1 | 1.8 | 55±0 | 1.8 | 70±0 | 1.8 |
| 3.4 | 10±1 | 3.4 | 33±1 | 3.1 | 59±1 | 2.6 | 73±1 | 2.4 | 86±0 | 2.4 |
| 4.4 | 10±2 | 4.4 | 34±1 | 3.6 | 64±1 | 2.7 | 73±1 | 2.5 | 92±2 | 2.5 |
| 5.0 | 7±1 | 4.9 | 30±2 | 3.9 | 71±2 | 2.9 | 75±1 | 2.7 | 93±2 | 2.7 |
| 5.5 | 11±2 | 5.3 | 30±1 | 4.3 | 63±2 | 3.0 | 72±1 | 2.8 | 89±1 | 2.7 |
| 6.0 | 8±0 | 5.6 | 28±2 | 4.3 | 52±2 | 3.1 | 73±1 | 2.9 | 89±1 | 2.8 |
| 6.8 | 10±2 | 5.9 | 41±5 | 4.0 | 69±1 | 3.3 | 77±0 | 3.2 | 82±4 | 3.1 |

TABLE II

GROWTH OF *Aspergillus niger* IN BREAD MEDIUM

| Acidity of medium | Time of growth, *hours* | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| | 41 | | 66 | | 93 | | 216 | |
| Initial pH | Dry matter | pH of medium | Dry matter | pH of medium | Dry matter | pH of medium | Dry matter | pH of medium |
| | *mgs.* | | *mgs.* | | *mgs.* | | *mgs.* | |
| 4.6 | 7±0 | 4.5 | 64±6 | 3.2 | 105±2 | 2.7 | 148±2 | 2.6 |
| 5.0 | 7±1 | 4.8 | 71±1 | 3.3 | 108±4 | 2.8 | 151±2 | 2.7 |
| 5.4 | 11±2 | 5.1 | 74±1 | 3.4 | 114±3 | 2.8 | 152±1 | 2.7 |
| 5.8 | 12±3 | 5.2 | 73±4 | 3.5 | 114±1 | 2.9 | 152±2 | 2.7 |
| 6.2 | 10±1 | 5.5 | 68±4 | 3.5 | 107±6 | 2.9 | 151±3 | 2.7 |

It is apparent that the rate of growth, as measured by the amount of dry matter produced, is not significantly effected by initial pH within the normal range of bread-making or between pH 4.5 and 6.8. Growth is appreciably retarded at pH 1.9 and below. The upper limit of growth was not determined. It is to be noted that during growth the pH shifts sharply in an acid direction, finally approaching nearly the same value in all cases. This value is probably determined principally by the amount of carbohydrate present in the medium. The development of the characteristic black conidia closely paralleled the rate of growth in all cases.

## Experimental Procedure

### Part II

### *The Specific Effect of Acids*

After it was determined that no sharp pH optimum existed, attention was directed to a study of the specific influence of several acids which are used to control "rope" in commercial bread-making.

Preliminary qualitative experiments indicated that acetic acid, in particular, has a marked specific influence on the growth of *Aspergillus niger*. For example, complete inhibition of growth was obtained in bread medium set at pH 3.5, and containing 0.2% acetic acid.

An experiment was then designed to compare several acids at identical pH levels. The technique used in this experiment was as follows:

50 cc. portions of bread medium set at pH 3.5 were sterilized in 200 cc. Erlenmeyer flasks. A definite quantity of the particular acids, also at pH 3.5, was then added. Then sterile water was added in amount sufficient to bring the total volume to 59 cc. Finally, the inoculum was added, thus making the final volume 60 cc.

The sterile solutions of the acids at pH 3.5 were prepared by setting the pH with sodium hydroxide. They were then made up to a definite volume and sterilized in glass-stoppered pressure bottles at 20 pounds for 20 minutes. The bread medium was prepared as described in Part I.

In this manner were prepared, in duplicate, flasks containing calcium acid phosphate, tartaric, phosphoric, lactic, citric, and acetic acids [2] in concentrations of 0.2%, 0.4%, and 0.6%. An appropriate control with no added acid was also prepared. Figure 1 is a photo-

[2] The acids used were the following:

Acetic —Merck's Blue Label 99.5%
Citric —Eimer & Amend T.P.
Lactic —Eimer & Amend C.P.
Phosphoric—Mallinkrodt Analytical Reagent 85%
Tartaric —Eimer & Amend T.P.
Calcium Acid Phosphate—Eimer & Amend T.P.

graph of the flasks, of the 0.2% and 0.6% series, after five days incubation at 30° C. The marked specific influence of acetic acid is clearly shown. Observations at the end of 2, 3, and 5 days in-

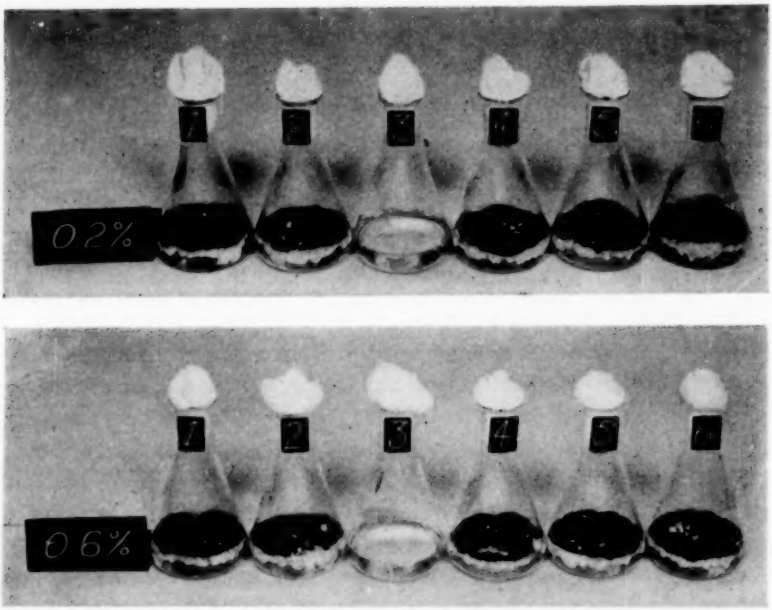



Fig. 1. Photograph of flasks of the 0.2% and 0.6% series after five days incubation. (1) Lactic acid, (2) Citric acid, (3) Acetic acid, (4) Tartaric acid, (5) Phosphoric acid, (6) Calcium acid phosphate.

cubation did not reveal any stimulation or inhibition of growth in any case except that of acetic acid. Complete inhibition was obtained with as little as 0.2% acetic acid, whereas even 0.6% of the other acids failed to cause even slight inhibition. This experiment was performed on a percent basis rather than a molar basis, but the extension of the range to 0.6% covers the difference in molarities.

## Experimental Procedure

### Part III

### *Specific Effect of Acetic Acid as a Function of Hydrogen-ion Concentration*

A series of experiments was made to determine the effect of acetic acid at different pH levels. The technique used, the setting of the bread medium, and the preparation of acetic acid solutions were carried out as described in Part II. The acetic acid concentration was 0.2% in all cases except the control where no acetic acid was used. Crops were harvested in triplicate, as in Part I.

From the results tabulated in Table III, it is apparent that the toxicity of acetic acid is dependent, at fixed concentration, on the initial pH of the medium. At pH 3.6 and below, 0.2% acetic acid is extremely toxic, as shown by the fact that only traces of growth were obtained in 162 hours. At pH 4.6 and above, the toxicity diminishes. Although, even here it is quite pronounced in the early stages of growth. Unfortunately, there was not included, in this experimental series, acetic acid-free controls at any pH other than 2.7.

TABLE III

EFFECT OF INITIAL PH UPON THE TOXICITY OF ACETIC ACID TOWARDS *Aspergillus niger*

| Acidity of medium | Concentration of ($CH_3COOH$) | Time of growth, *hours* | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| | | 47 | | 69 | | 96 | | 162 | |
| Initial pH | | Dry matter | pH of medium | Dry matter | pH of medium | Dry matter | pH of medium | Dry matter | pH of medium |
| | | *mgs.* | | *mgs.* | | *mgs.* | | *mgs.* | |
| 2.7 | 0 | 51∓1 | 2.6 | 90∓1 | — | 122±2 | 2.1 | 131±2 | 2.2 |
| 2.7 | 0.2% | No growth | 2.6 | No growth | — | No growth | — | No growth | 2.5 |
| 3.6 | 0.2 | No growth | 3.6 | No growth | — | Trace | — | 3+1 | 3.5 |
| 4.6 | 0.2 | 26±4 | 4.6 | 85±2 | 4.2 | 119±4 | 3.3 | 143±2 | 2.8 |
| 5.0 | 0.2 | 33±2 | 5.0 | 90±1 | 4.6 | 116±1 | 3.6 | 150±3 | 3.0 |
| 5.6 | 0.2 | 44±1 | 5.0 | 87±3 | 4.9 | 116±1 | 3.8 | 143±3 | 3.0 |
| 6.0 | 0.2 | 41±2 | 5.5 | 81±2 | 5.1 | 106±1 | 3.9 | 142±2 | 3.0 |
| 6.4 | 0.2 | 43±2 | 5.2 | 83±3 | 5.1 | 110±2 | 4.0 | 140±2 | 3.0 |
| 7.0 | 0.2 | 34±2 | 5.9 | 76±2 | 5.2 | 105±2 | 4.3 | 134±1 | 3.0 |

The effect of acetic acid was further studied at pH levels of about 5.0, 5.5 and 6.0. Acetic acid was used in concentrations of 0.2% and 0.4%. Acetic acid-free controls were made at each of the above pH levels. The results are summarized in Table IV. Definite inhibition of growth was obtained in every instance where acetic acid was used. Furthermore and of equal consideration is the fact that, for the same concentration of acetic acid, the degree of inhibition increases with increasing H-ion concentration; while at the same initial pH, greater inhibition was obtained with increasing concentration of acetic acid. These particular results are of especial interest, not only from practical considerations but also from a purely biochemical viewpoint. They quite clearly indicate that the toxicity is due to the undissociated acetic acid. It has already been shown (Part I) that hydrogen-ion concentration alone does not greatly influence the growth of the fungus, especially within the pH range of 4.5 to 6.5.

TABLE IV

EFFECT OF pH ON THE GROWTH OF *Aspergillus niger* IN THE PRESENCE AND ABSENCE OF ACETIC ACID

| Acidity of medium | Concentration of ($CH_3COOH$) | Time of growth, *hours* | | | | | |
|---|---|---|---|---|---|---|---|
| | | 42 | | 66 | | 92 | |
| Initial pH | | Dry matter | pH of medium | Dry matter | pH of medium | Dry matter | pH of medium |
| | | *mgs.* | | *mgs.* | | *mgs.* | |
| 5.2 | 0 | 39±1 | 4.4 | 100±1 | 3.2 | 124±1 | 2.9 |
| 5.1 | 0.2% | 12±1 | 5.1 | 70±4 | 4.8 | 115±2 | 3.7 |
| 5.0 | 0.4% | 7±4 | 5.0 | 30±2 | 5.1 | 96±2 | 5.1 |
| 5.5 | 0 | 37±1 | 4.6 | 94±1 | 3.3 | 121±3 | 2.9 |
| 5.5 | 0.2% | 22±2 | 5.3 | 74±3 | 5.0 | 105±1 | 4.1 |
| 5.5 | 0.4% | 13±2 | 5.5 | 57±0 | 5.4 | 101±6 | 5.1 |
| 6.1 | 0 | 34±2 | 4.8 | 96±3 | 3.4 | 121±3 | 3.0 |
| 6.0 | 0.2% | 29±5 | 5.6 | 76±3 | 5.1 | 102±1 | 3.9 |
| 6.0 | 0.4% | 21±3 | 5.7 | 71±3 | 5.5 | 100±3 | 5.0 |

## Experimental Procedure

### PART IV

*Experiments with Bread Loaves*

Efforts were next directed to a study of the effect of acetic acid, in the form of 90 grain "distilled" vinegar, on the development of *Aspergillus niger* on the bread loaf itself.

As well as could be determined, observations concerning the molding of bread have usually been made in the following manner:

The bread is cooled in an open room, then wrapped and allowed to incubate at any convenient temperature, usually not constant nor reproducible. Reliance is placed on chance infection.

Such procedure supplies statistical data of a sort, but could hardly be called an accurate or reliable method. It was decided in the present investigation to leave little to chance and to work under conditions which could be exactly reproduced in any laboratory.

A series of pup loaves was baked in a revolving oven. These loaves were made in duplicate. The same formula was used in all cases with the exceptions noted below:

| | pH of loaf |
|---|---|
| Series 1, No vinegar added | 5.5 |
| " 2, ½% of 90 grain vinegar added | 5.2 |
| " 3, 1% " " " " " | 5.1 |
| " 4, 2% " " " " " | 4.9 |

These loaves were transferred from the oven to sterile bell jars by means of sterile tongs. After they were allowed to cool overnight, they were aseptically transferred to a sterile wooden box. This box was fitted with four port holes, one on each of the four sides, near the top.

The loaves were placed on a shelf in the box, with the break side downward. A suspension of *Aspergillus niger* conidia was atomized through each of the port holes in turn. The loaves were then placed under bell jars and kept at 30° C. The air under the bell jars was kept saturated with moisture by an open dish of water. Observations were made from time to time upon the relative amounts of mold growth.

TABLE V

GROWTH OF MOLD ON BREAD LOAVES—EFFECT OF VINEGAR

| Experiment number | I | | II | | III | | IV | | V | | VI | | VII | | Totals |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | A | B | A | B | A | B | A | B | A | B | A | B | A | B | |
| No vinegar | 4 | 4 | 3 | 2 | 4 | 2½ | 4 | 3½ | 4 | 3½ | 2½ | 2½ | 4 | 3½ | 47 |
| ½% vinegar | 2 | 3 | 4 | 3 | 3 | 4 | 3 | 2 | 2½ | 2 | 2½ | 4 | 3 | 3½ | 41½ |
| 1% vinegar | 3 | 2 | 2 | 4 | 2 | 2½ | 2 | 3½ | 2½ | 3½ | 4 | 2½ | 2 | 2 | 37½ |
| 2% vinegar | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 14 |

The scoring system used in making these comparisons is much the same as is sometimes used in scoring bread for flavor and odor. The loaf, in a series of four, which showed the heaviest growth of mold was assigned the value 4. Next in order was marked 3, then 2, and finally, the loaf showing the least mold was given the value 1. If no choice could be made between the two showing the least mold, then each was assigned the value 1½. Similarly, for the two showing the most mold, the score was 3½, and for the two intermediate the score was 2½.

The results as shown in Table IV represent 14 sets of experiments, two sets being done at one time. The slight irregularity, which may be observed, is probably due to small differences in baking.

Examination of the results shows that acetic acid, in the form of vinegar, definitely inhibits the growth of *Aspergillus niger*. For "rope" control, vinegar is usually used at ½% to 1% concentration. The results indicate that, even at these concentrations, there is some inhibition if the pH of the bread is in the neighborhood of 5.0 to 5.2. Other experimental data showed that higher concentrations of vinegar

were necessary to produce inhibition in those cases where the formulae were such as to finish with bread having higher pH values than these. Stimulation of mold growth, by the addition of vinegar or by increasing the hydrogen-ion concentration of doughs, is not possible on the basis of results herein reported.

## Summary

In liquid bread medium, *Aspergillus niger* exhibits no well-defined growth optimum with respect to hydrogen-ion concentration. This mold was found to develop equally well at several pH levels, which embrace the pH range within which commercial bread is produced.

Calcium acid phosphate, tartaric, phosphoric, lactic, and citric acids in concentrations of 0.2%, 0.4% and 0.6% were found to have no apparent effect upon the growth and development of *Aspergillus niger* in liquid bread medium at pH 3.5. Under the same conditions, acetic acid in concentrations of 0.2%, 0.4% and 0.6% was found to inhibit completely the growth of *Aspergillus niger*.

The toxicity of acetic acid toward *Aspergillus niger* was found to depend upon the pH of the culture medium. The higher the hydrogen-ion concentration, the lower is the concentration of acetic acid necessary to inhibit completely the growth of the fungus.

90 grain vinegar, added to doughs in the amounts of ½%, 1%, and 2% of the quantity of flour, was found to have an inhibiting action upon the development of *Aspergillus niger* on the finished baked loaves of bread.

In conclusion, this study indicates that the growth of *Aspergillus niger* on bread would not be stimulated or favored by the addition of acid substances to dough for the purpose of controlling "rope."

The effect of acidity on the growth of other molds is under investigation.

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# EFFECT OF DIFFERENT BUFFERS AND TYPE OF SUBSTRATE ON THE DIASTASIS OF WHEAT STARCH [1, 2]

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Probably the most important chemical change occurring in bread doughs during the fermentation period is the conversion of starch to sugar. Conversion of starch to sugar by diastatic enzymes is a process commonly used in many industries, but the conversion of starch to sugar in fermenting doughs differs in one very important respect from the process used in most fermentation industries. In bread doughs, the starch is in the raw or natural state, while in most fermentation processes, the starch is cooked before addition of the diastatic enzyme.

For most of the work on diastatic enzymes cooked starch has been used as the substrate, and in many cases, the substrate used is a cooked soluble starch, similar to Lintner's soluble starch. Results obtained by using such materials are not necessarily applicable, therefore, when raw starch is used as the substrate.

Starch granules, as found in nature, are insoluble in cold water, but upon treatment with boiling water become gelatinized and form a paste. Wheat starch according to Alsberg (1926, 1927) does not disintegrate when boiled to form a truly colloidal solution. The granules merely swell to form a suspension of swollen granules floating more or less freely in the watery phase.

It is generally agreed that starch granules consist of two separate chemical constituents, namely, a water insoluble material, which is designated as amylopectin or $\alpha$-amylose, which forms the outer part or envelope of the granule, and a water soluble constituent designated as amylose or $\beta$-amylose which is found in the interior of the granule. The amylopectin or $\alpha$-amylose constituent is not soluble in boiling water, but evidently becomes highly hydrated and possibly undergoes some chemical change when treated with boiling water.

Starch in flour may be presumed to be in the natural state, excepting possible mechanical injury to the granules during the milling process. Alsberg and Perry (1924) found 60% of wheat starch was soluble in cold water after grinding 122 hours in a pebble mill with flints. This

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[2] Contribution from Department of Cereals and Milling, North Dakota Agricultural Experiment Station, Fargo, North Dakota.

would indicate that some mechanical injury to granules is not only possible, but probable, in the ordinary milling process. Shollenberger and Coleman (1926) found that overgrinding of flour increased diastatic activity and the same result is reported by Karacsonyi and Bailey (1930) and Markley and Bailey (1934). In the ordinary milling process, however, it is probable that most of the starch granules escape mechanical injury, but the small portion which suffer mechanical injury may have an important effect on the diastatic activity and other properties of the flour.

When pure starches are prepared from wheat flour, the broken or injured granules are entirely removed in the purification process—in our experience. Four different types of substrates are therefore possible in working with diastatic enzymes: (1) Lintner's soluble starch dissolved in boiling water, (2) ordinary starch treated with boiling water, (3) natural starch in flour which may consist partly of mechanically injured granules, and (4) purified wheat starch consisting of uninjured granules. In the case of flour, we also have present substances such as proteins, sugars, and lipoids, which may affect diastatic activity.

Diastatic enzymes are found in both plants and animals. The action of diastase on starch has received considerable study. Waksman and Davidson (1926) suggest that the diastatic conversion of starch to sugar in bread doughs consists of a series of interdependent reactions, and it is reasonable to presume that diatase contains at least two components, (1) an amylase, which hydrolyzes starch to dextrin and (2) a dextrinase which converts the dextrin to maltose. Ohlsson (1930) and Nordh and Ohlsson (1932) have devised a method of separating the two components. The $\alpha$-amylase or dextrinogenase is acid-labile and is inactivated at pH 3.3, while the $\beta$-amylase or saccharogenase is thermo-labile and is inactivated by heating to 70° C. for 15 minutes.

Baker and Hulton (1914) found an abundance of saccharifying enzyme in wheat flours, but found that some flours are lacking in the liquifying fraction. Andrews and Bailey (1934) report that flour from sound wheat contains mostly the $\beta$-amylase or saccharifying fraction, while flour from sprouted wheat contains considerable of the $\alpha$-amylase or liquefying fraction.

Ford and Guthrie (1908) in their studies on diastatic power of flour extracts found that the diastatic activity varied inversely as the time of extraction. Use of phosphate and potassium chloride solutions as extracting agents gave extracts of higher diastatic activity than water. Ford and Guthrie ascribe this to protecting influence of the electrolytes, but Jozsa and Gore (1932) suggest that increased solubility of the enzyme in the salt solutions is responsible.

Gore and Jozsa (1932) conclude that neither the liquefying power

nor the saccharifying power of diastase, using starch paste or a solution of soluble starch as substrate, has any direct bearing on the effective diastatic properties of flour.

Baker and Hulton (1908) examined barley starch digested with precipitated malt diastase for varying lengths of time. Maltose was the only sugar present when the period of digestion was relatively short, but dextrose was found after long digestion. Only 80% of the liquefied products were fermentable by yeast and the dextrins isolated from the solution were quite resistant to attack by diastase. This would indicate that complete conversion to sugar does not occur.

The effect of pH on diastatic activity has been studied using as substrate soluble starch or cooked starch. Sherman and Thomas (1915) find an optimum range of 4.2–4.6 for malt diastase using soluble potato starch. Sherman, Thomas, and Baldwin (1919) narrowed the optimum range to 4.5–4.6 for malt diastase and to 4.8 for taka-diastase.

Sherman and co-workers (1927, 1930) found that the optimum pH for action of malt diastase on starch tended to shift when the temperature and time of digestion was varied. The optimum pH for 30 minutes digestion was 4.6 as compared with 4.8 for 2 hours digestion. The diastase is more sensitive to acidity as temperature increases. This confirmed the results of Olsen and Fine (1924) who found a shift in the optimum pH with change of temperature. Sherman and co-workers found that optimum pH changes when the concentration of salt or buffer solution is varied.

Rumsey (1922) found that wheat diastase had approximately the same optimum pH as barley malt at 27° C. He found the optimum pH to be 4.7–4.8 with a broader range of maxima between 4.0 and 5.3. Rumsey developed a method for determining diastatic activity of flour, but did not attempt to control pH during digestion. Later methods have controlled pH by use of buffer solutions. Malloch (1929) used a citrate buffer while the Blish-Sandstedt method (1933) uses an acetate buffer solution, both having a pH range of 4.6 to 4.8.

## Experimental

### *Effect of Different Buffer Solutions on Diastatic Activity of Flour*

Three buffer solutions of rather different composition were used for this study, namely:

(1) McIlvaine's citric acid-sodium phosphate buffer, pH range 2.2–8.0.

(2) Walpole's acetate buffer, pH range 3.6–5.6.

(3) Sørenson's phosphate buffer, pH range 5.3–8.0.

The buffers were prepared according to directions given by Clark (1925).

In determining the diastatic activity of the flour 46 cc. of the required buffer solution was added to 5 g. of patent flour, similar to the Blish-Sandstedt procedure, and digestion allowed to proceed 60 minutes at 30° C. The Blish-Sandstedt procedure was then followed with the exception that with buffers of pH 5.0 or above it was necessary to add a few drops of concentrated $H_2SO_4$ to insure precipitation of proteins by sodium tungstate. Sufficient concentrated $H_2SO_4$ was added to give a red color with thymol blue.

The results are given in Table I and in Figure 1. Results in this case are given on basis of milligrams of maltose formed from 5 g. of flour rather than the customary Rumsey units based on 10 g. of flour.

TABLE I

EFFECT OF pH ON DIASTASIS OF FLOUR

Comparison of different buffer solutions

| pH | McIlvaine's citric acid-sodium phosphate buffer | Walpole's acetate buffer | Sørenson's phosphate buffer |
|---|---|---|---|
| | *Mg. maltose* [1] | *Mg. maltose* [1] | *Mg. maltose* [1] |
| 3.0 | 45.8 | | |
| 3.4 | 81.0 | | |
| 3.6 | — | 112.3 | |
| 4.0 | 102.6 | 109.8 | |
| 4.6 | — | 110.0 | |
| 5.0 | 116.5 | 107.7 | |
| 5.2 | — | — | 133.2 |
| 5.6 | — | 108.6 | 133.4 |
| 6.0 | 85.0 | | 130.8 |
| 6.4 | — | | 128.4 |
| 6.6 | 75.2 | | |
| 7.0 | 68.8 | | 117.8 |
| 8.0 | 53.4 | | 99.5 |

[1] From 5 g. of flour.

The largest maltose production occurred with Sørenson's phosphate buffer, and indicates that this buffer offers more favorable conditions for maltose production than the other two buffers.

McIlvaine's citric acid-sodium phosphate buffer shows a sharper maximum than the other two buffers with an optimum point at approximately pH 5.0. The optimum point for Sørenson's phosphate buffer lies between pH 5.2 and 5.6. The results with Walpole's acetate buffer were rather surprising, since no definite optimum point is shown throughout the pH range of this buffer.

The optimum point for McIlvaine's buffer and Sørenson's phosphate buffer differ by 0.5 pH, but at pH 8.0 with Sørenson's phosphate buffer 99.5 mg. maltose were formed as compared with 53.4 mg. maltose with McIlvaine's buffer. Diastatic activity is apparently less sensitive to

change in pH with either Walpole's or Sørenson's buffer as compared with the McIlvaine buffer. The proteins of the flour are probably responsible for the peculiar results obtained with the Walpole buffer,

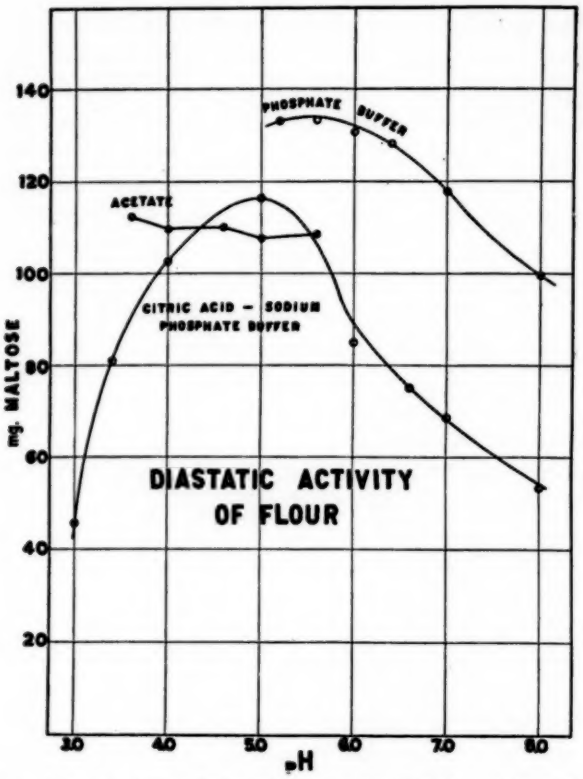



Fig. 1. Effect of different buffer solutions on the diastatic activity of flour.

since, in studies following, using pure starch as the substrate, the acetate buffer does not show this peculiarity.

*Effect of Different Buffer Solutions on Diastasis of Raw Starch*

A sample of purified hard red spring wheat starch prepared in this laboratory was used as the substrate, and Merck's malt diastase and Parke Davis' taka-diastase used as source of the enzyme. The procedure was as follows:

The equivalent of 5 g. of dry starch was weighed into an Erlenmeyer flask and 25 mg. of the enzyme preparation added. Then the required buffer solution was added and same procedure followed as with flour. Results are reported on basis of milligrams of maltose formed from 5 g. dry starch. The results are given in Tables II and III and in Figure 2.

The values for malt diastase (Table II), it will be noted, are somewhat lower than those obtained with flour using natural flour diastase, but are of the same order of magnitude. The Walpole and Sørenson buffers show only a slightly wider optimum pH range than the citric acid-sodium phosphate buffer.

TABLE II

EFFECT OF DIFFERENT BUFFERS ON ACTION OF MALT DIASTASE ON RAW STARCH

| pH | McIlvaine's citric acid-sodium phosphate buffer | Walpole's acetate buffer | Sørenson's phosphate buffer |
|---|---|---|---|
| | *Mg. maltose* [1] | *Mg. maltose* [1] | *Mg. maltose* [1] |
| 3.0 | 0 | | |
| 3.6 | — | 13.2 | |
| 4.0 | 18.5 | 21.1 | |
| 4.2 | 25.8 | 28.3 | |
| 4.4 | 35.4 | 38.9 | |
| 4.6 | 44.2 | 49.9 | |
| 4.8 | 56.6 | 75.4 | |
| 5.0 | 66.9 | 86.9 | |
| 5.2 | 78.4 | 97.0 | 99.5 |
| 5.4 | 89.2 | — | — |
| 5.6 | — | 99.1 | — |
| 5.8 | 94.2 | | — |
| 6.0 | — | | 104.3 |
| 6.2 | 92.8 | | — |
| 6.4 | — | | 97.7 |
| 6.6 | 91.0 | | 95.3 |
| 7.0 | 82.1 | | 92.1 |
| 7.4 | — | | 75.9 |
| 8.0 | 50.2 | | 56.8 |

[1] From 5 g. of raw starch.

The optimum point when using McIlvaine's as well as Sørenson's phosphate buffer solutions is approximately at pH 6.0, while the optimum point for Walpole's buffer solution lies between pH 5.0 and pH 6.0. The optimum range for Walpole's buffer is not so wide in this case as with flour, which would indicate that the substances present in flour such as proteins have a tendency to increase the optimum range The optimum range for Sørenson's phosphate buffer is affected in the same manner but to much lesser degree.

Table III gives the results with taka-diastase. The taka-diastase values are much lower than the results obtained with malt diastase. While the taka-diastase used was diluted with sugar (indicated by blank determination) the dilution will not account for the great difference in maltose formation by equivalent weights of the two preparations.

As in the case with malt diastase, Walpole's acetate buffer and Sørenson's phosphate buffer show somewhat greater maltose formation than the determinations with McIlvaine's buffer at optimum points. Taka-

diastase apparently has a rather broader optimum range especially on the acid side than malt diastase.

The optimum pH for taka-diastase is more acid than that of malt diastase. For Walpole's buffer, the highest value was obtained at pH

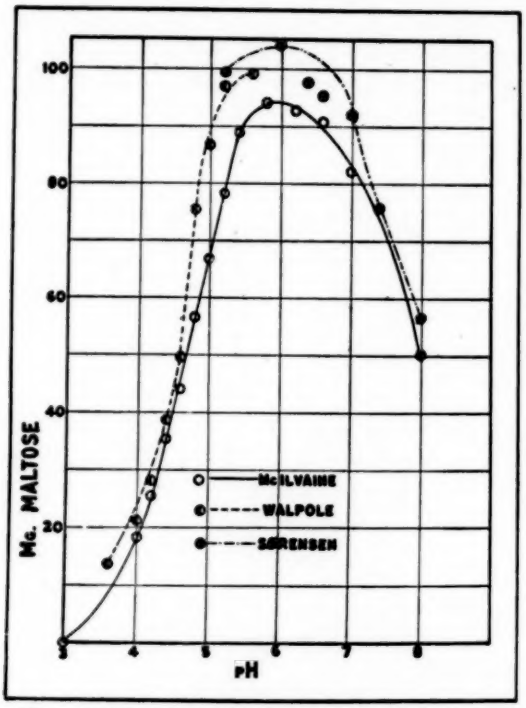



Fig. 2. Effect of different buffer solutions upon the diastasis of raw starch.

TABLE III

EFFECT OF DIFFERENT BUFFERS ON ACTION OF TAKA-DIASTASE ON RAW STARCH

| pH | Buffer Used | | |
|---|---|---|---|
| | McIlvaine's citric acid-sodium phosphate buffer | Walpole's acetate buffer | Sørenson's phosphate buffer |
| | *Mg. maltose* [1] | *Mg. maltose* [1] | *Mg. maltose* [1] |
| 3.0 | 5.5 | | |
| 3.6 | — | 10.0 | |
| 3.8 | — | 13.2 | |
| 4.0 | 16.0 | 15.0 | |
| 4.2 | — | 19.1 | |
| 4.4 | — | 23.2 | |
| 4.6 | — | 26.3 | |
| 5.0 | 22.2 | 30.3 | |
| 5.2 | — | — | 29.7 |
| 5.6 | — | 29.8 | — |
| 6.0 | 23.0 | — | 25.1 |
| 7.0 | 19.6 | — | 18.6 |
| 8.0 | 10.7 | — | 8.0 |

[1] From 5 g. of raw starch.

5.0, and Sørenson's buffer gave the highest value at pH 5.2—the limit of the acid range of this buffer.

The variation in maltose formation due to the type of buffer, however, is not great. With both malt and taka-diastase, the diastatic activity with the three buffers is of the same order of magnitude.

It is interesting to note, however, that when malt diastase is used on pure starch as the substrate, the optimum pH as compared with that for flour with natural flour diastase, has in each case shifted to the alkaline side. Assuming from Rumsey's results (1922) that the flour diastase and malt diastase are similar in their reaction to H-ions, this would indicate a definite protective action of the proteins at the higher H-ion concentrations. The sugar naturally present in the flour at the start of the reaction may also have some protective influence.

The results indicate, therefore, that whenever we change the environment of the diastatic enzyme in any manner, we are likely to have different optimum points for different factors such as pH.

### *Effect of pH Using Cooked Starch as the Substrate*

In the preceding studies raw or natural purified starch was used as the substrate. The results with cooked starch are of interest in showing the importance of the substrate.

A 1% starch paste was used as the substrate and the procedure was as follows: An approximately 2% starch paste was prepared by treating a weighed portion of the pure hard red spring wheat starch with boiling water and maintaining at this temperature for 30 minutes. After cooling the paste was diluted with distilled water so that it contained 2% starch by weight. Double-strength McIlvaine's buffer solutions for the required pH were prepared and to 23 cc. each of the starch paste and buffer were added 25 mg. of malt diastase or taka-diastase. The 46 cc. of solution therefore contained 460 mg. of starch as compared with 5000 mg. of raw starch used as substrate.

The results are given in Table IV and in Figure 3. The optimum pH, as compared with results using raw starch as the substrate, has shifted to the acid side. Malt diastase shows a broader optimum pH range when cooked starch is used as the substrate and pH 5.0 and 6.0 give approximately the same value. For taka-diastase a rather definite optimum point at pH 5.0 is noted. Malt diastase is more tolerant to alkali and much less tolerant to acid than taka-diastase.

One hour digestion with cooked starch as the substrate produces a much greater quantity of maltose than when raw starch is used as the substrate. While sugar formation is somewhat lower when taka-diastase is used as the enzyme the results are of same order of magnitude

as those obtained with malt diastase. The difference in potency of the two enzyme preparations is much less, therefore, when cooked starch is used as the substrate, since with raw starch malt diastase produced 3 to 4 times as much sugar as taka-diastase.

TABLE IV

EFFECT OF pH ON DIASTATIC ACTIVITY WITH COOKED STARCH AS SUBSTRATE

| pH | Maltose formed from 46 cc. 1% starch paste [1] | |
|---|---|---|
| | Malt diastase | Taka-diastase |
| | *Mg.* | *Mg.* |
| 2.2 | 2.8 | — |
| 2.8 | 4.4 | 15.1 |
| 3.0 | 18.2 | 26.0 |
| 3.2 | 125.0 | 48.2 |
| 3.4 | 240.5 | 64.4 |
| 3.6 | 286.0 | 89.7 |
| 4.0 | 311.3 | 279.0 |
| 5.0 | 319.4 | 296.1 |
| 6.0 | 320.0 | 261.1 |
| 7.0 | 307.7 | 199.1 |
| 8.0 | 282.1 | 57.0 |

[1] 46 cc. of starch paste contained 460 mg. starch. 25 mg. diastase preparations used with McIlvaine's citric acid-sodium phosphate buffer.

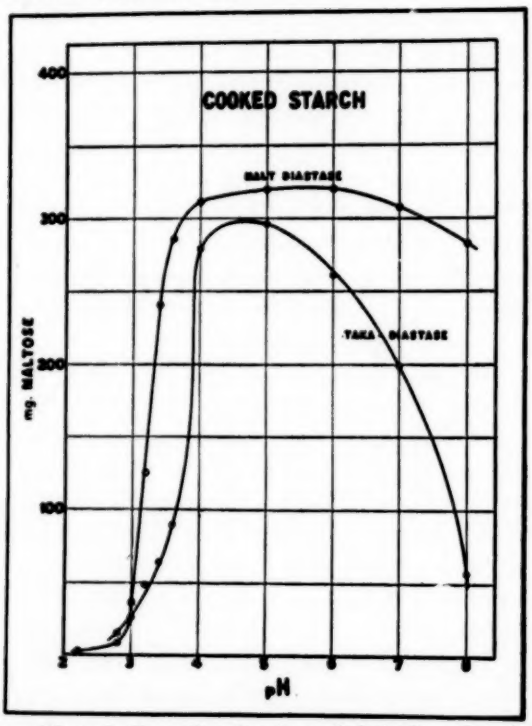



Fig. 3. Effect of pH on diastasis using cooked starch as the substrate.

With malt diastase more than 3 times as much sugar was formed from cooked starch than from raw starch and results with taka-diastase show a much greater variation. Figure 4 records a comparison of results with raw and cooked starch using malt diastase as source of enzyme.

If we consider the variation between cooked and raw starch in terms of percentage of the substrate converted to maltose, the difference becomes much larger. Only 460 mg. of cooked starch was used as substrate compared to 5000 mg. of raw starch. With cooked starch a weight of maltose equivalent to 60 to 70% of the weight of starch was produced, and with the same quantity of malt diastase only about 2% of the raw starch substrate was converted to sugar.

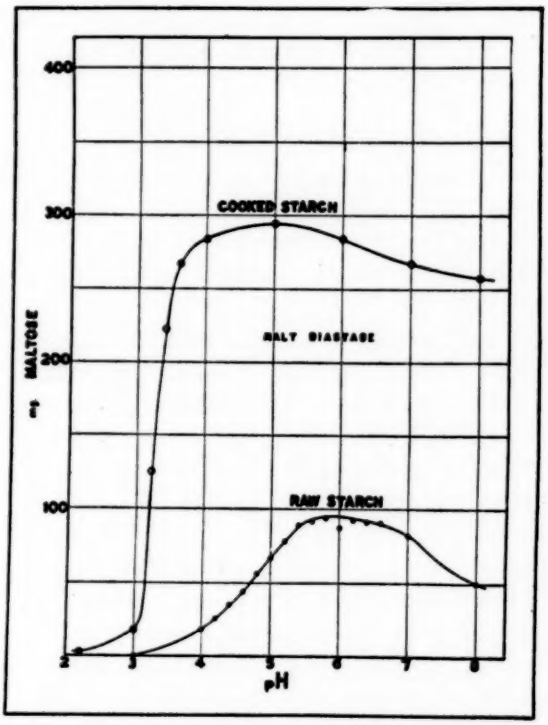



Fig. 4. Comparison of diastasis of cooked and raw starch at variable pH.

## Discussion

Wheat flour and pure raw starch as substrates gave results of the same order of magnitude. The difference noted between raw and cooked starch as substrates would indicate that results obtained using a

cooked or soluble starch as the substrate are of practically no value in predicting the diastatic susceptibility of raw starch.

In any work pertaining to the diastatic activity of wheat flour doughs, it is quite important that the starch substrate be similar to the natural starch of the wheat flour. In sugar formation from raw starch the limiting factor apparently is the ability of the enzyme preparation to liquefy or render soluble the raw starch. The $\alpha$-amylase or liquefying fraction is the most important consideration since the $\beta$-amylase or saccharifying fraction is apparently present in abundance in most cases.

The optimum pH for diastatic activity will vary with conditions of the substrate and other environment. The shift due to cooking the starch is explainable, since from the work of Ohlsson, the $\alpha$-amylase or liquefying fraction is acid-labile, and this fraction is the limiting factor with raw starch. The shift of optimum pH noted in flour is very probably due to the protective action of proteins, sugar, and other flour ingredients.

## Summary

Wheat flour produced more maltose in 60 minutes with Sørenson's phosphate buffer, than with McIlvaine's or Walpole's buffer. McIlvaine's buffer showed an optimum at pH 5.0, Sørenson's buffer pH 5.2–5.6 and Walpole's buffer did not show an optimum point with flour as the substrate.

Using pure raw starch as substrate and malt and taka-diastase as source of enzyme, the optimum pH tends to shift to the alkaline side. The optimum for McIlvaine's buffer shows greater shift than for Sørenson's phosphate buffer.

Using cooked starch as substrate and malt and taka-diastase as source of enzyme, the optimum pH as compared with raw starch tends to shift to the acid side.

Much greater maltose formation occurs when cooked starch is used as a substrate when compared with raw starch using equivalent amounts of enzyme preparation. With malt diastase 60 to 70% of the substrate is converted to sugar when cooked starch is the substrate, while the same quantity of enzyme will convert only 2% of the raw starch substrate under similar conditions.

The difference in sugar formation due to cooking the starch is so large as to indicate that results with cooked starch as substrate are of practically no value in predicting the activity of diastase on raw or natural starch.

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## FACTORS AFFECTING THE DIASTATIC ACTIVITY OF WHEAT FLOUR. II.[1]

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(Received for publication October 11, 1934)

In order to determine the relative diastatic activities of flours milled from different varieties of wheat it was found necessary to replicate the variety trials at more than one location in order to eliminate climatic and soil effects (Markley and Bailey, 1934). Also it was found that if means of at least three locations were used it was possible to predict with fair accuracy ($r = .69$) the diastatic activity of the patent flour from that of the wheat.

If blended samples could be used instead of individual determinations upon a large number of individual samples the labor required for varietal studies would be very much reduced. In the first portion of this study individual determinations and means for both flour and wheat diastatic activity of 13 varieties of spring wheat grown at 3 locations in Minnesota in 1933 were reported. In Table I of this paper the mean results are repeated. Equal portions of each of the flours of each variety were combined into varietal composites and the diastatic activity determined. The wheat samples were combined before grinding. The results are given in Table I. There was a very high order of agreement between the means of individual determinations and the determinations upon the composite samples as was evidenced by a correlation of $r_{AB} = .96$ in the case of the flours and $r_{CD} = .93$ in the case of the wheat meal samples. The absolute levels of diastatic activity of the composite samples were a little lower than that of the individual samples. This was probably due to the composite determinations being made about three months subsequent to the individual determinations.

For the purpose of predicting the diastatic activity of flour from an analysis of the wheat the compositing method seems to be as reliable as

[1] Published with the approval of the Director as Paper No. 1310, Journal Series, Minnesota Agricultural Experiment Station.

TABLE I

COMPARISON OF THE DIASTATIC ACTIVITIES OF COMPOSITE SAMPLES WITH THE MEANS OF DETERMINATIONS UPON INDIVIDUAL SAMPLES, 1933 CROP SPRING WHEAT

(Rumsey units)

| Variety number | Flour | | Whole wheat meal | |
|---|---|---|---|---|
| | Means of individual determinations *A* | Composite sample *B* | Means of individual determinations *C* | Composite sample *D* |
| 2610 | 170 | 164 | 266 | 226 |
| 2308 | 152 | 152 | 295 | 243 |
| 2223 | 205 | 192 | 318 | 292 |
| 2297 | 170 | 158 | 291 | 260 |
| 2315 | 167 | 165 | 285 | 254 |
| 2340 | 164 | 157 | 264 | 227 |
| 2621 | 161 | 157 | 270 | 242 |
| 2607 | 161 | 154 | 260 | 219 |
| 2303 | 174 | 166 | 284 | 254 |
| 2202 | 149 | 142 | 261 | 226 |
| 2309 | 153 | 142 | 273 | 237 |
| 2204 | 162 | 152 | 271 | 224 |
| 1239 | 150 | 138 | 274 | 240 |
| Mean | 164.46 | 156.85 | 277.85 | 241.85 |
| Standard deviation | 14.05 | 13.27 | 15.81 | 19.08 |
| Correlations | | | | |
| $r_{AB} = .96$ | $r_{AC} = .69$ | $r_{BD} = .72$ | $r_{CD} = .93$ | 5% point = .55<br>1% point = .68 |

the use of means of individual determinations, as is indicated by the correlation $r_{AC} = .69$ for the means of individual determinations and $r_{BD} = .72$ for the composite samples. These relationships might not hold true in the case of any of the samples being badly weathered, heat damaged, or partially germinated. The compositing of samples must be used only with sound samples.

The rate of sugar formation in buffered flour suspensions with digestion times from 1 to 8 hours was investigated by Landis (1934) and found to be a logarithmic function of the time. If this relation holds for periods of digestion of less than 1 hour it might be possible without too great a loss of accuracy to reduce the digestion time and still get relative values for diastatic activity. Such a procedure would be of value in mill control work particularly where supplementation of diastatic deficiencies is being practiced.

A series of 19 samples of wheat and patent flour experimentally milled from each was available for this study. These wheat samples were blends of equal quantities of wheat grown in the rod row nurseries at each of three stations in Minnesota during 1933. In making the diastatic activity determinations in the case of the flours, digestion pe-

TABLE II

AMOUNTS OF REDUCING SUGARS PRODUCED BY AUTOLYTIC DIGESTION OF PATENT FLOURS MILLED FROM DIFFERENT VARIETIES OF SPRING WHEATS, 1933 CROP, DURING VARYING DIGESTION PERIODS

(Rumsey units)

| Sample number | 15 Min. *E* | 30 Min. *F* | 60 Min. *G* | 120 Min. *H* |
|---|---|---|---|---|
| 15578 | 96 | 118 | 135 | 154 |
| 15579 | 80 | 95 | 116 | 126 |
| 15580 | 116 | 140 | 169 | 188 |
| 15581 | 92 | 110 | 128 | 137 |
| 15582 | 88 | 104 | 130 | 141 |
| 15583 | 113 | 130 | 157 | 179 |
| 15584 | 99 | 119 | 146 | 163 |
| 15585 | 178 | 214 | 246 | 276 |
| 15586 | 83 | 94 | 111 | 123 |
| 15587 | 97 | 114 | 139 | 156 |
| 15588 | 87 | 104 | 126 | 134 |
| 15589 | 97 | 116 | 134 | 146 |
| 15590 | 79 | 91 | 120 | 123 |
| 15591 | 92 | 111 | 129 | 137 |
| 15592 | 83 | 100 | 118 | 125 |
| 15593 | 85 | 100 | 120 | 139 |
| 15594 | 84 | 94 | 118 | 126 |
| 15595 | 95 | 109 | 132 | 135 |
| 15596 | 65 | 78 | 94 | 99 |
| Mean | 95.21 | 112.68 | 135.16 | 147.74 |
| Standard deviation | 22.59 | 27.69 | 30.73 | 36.53 |

Correlations

$r_{EF} = .996$ $r_{EG} = .993$ $r_{EH} = .984$ $r_{FG} = .992$ $r_{FH} = .985$

$r_{GH} = .989$ 5% point = .456 1% point = .575

riods of 15, 30, and 120 minutes were used in addition to the standard 60-minute period. Blank determinations were made upon the flour samples and all flour samples given in Table II were corrected for blanks. In the case of the wheat samples quantities were not sufficient for the blank determinations and for the 30-minute digestion period.

In any of the four digestion periods the placing of the varieties was essentially the same since the correlations between the different digestion periods were all $r = .98$ or higher. It is of interest to note that 70% of the sugar formed during 60 minutes digestion was converted during the first 15 minutes, and 83% during the first 30 minutes. Prolonging the digestion period for a second hour only resulted in an average increase of 9% over the first hour. When the weight of sugar formed was plotted against the logarithm of the time, the points tended to fall upon straight lines, with greater slopes in the case of the higher diastatic samples. This is in accord with the findings of Landis (1934). There would be little gain in accuracy in this method of determining diastatic activity by prolonging the digestion period to 2 hours, and for

rapid work it should be possible in the light of these observations to shorten the digestion period to 30 minutes without a serious increase in analytical errors.

TABLE III

AMOUNTS OF REDUCING SUGARS PRODUCED BY AUTOLYTIC DIGESTION OF FINELY GROUND MEAL OF DIFFERENT VARIETIES OF SPRING WHEATS, 1933 CROP, DURING VARYING DIGESTION PERIODS

(Rumsey Units)

| Sample number | 15 Min. *I* | 60 Min. *J* | 120 Min. *K* |
|---|---|---|---|
| 15578 | 173 | 240 | 263 |
| 15579 | 164 | 220 | 279 |
| 15580 | 206 | 290 | 335 |
| 15581 | 181 | 260 | 300 |
| 15582 | 178 | 256 | 315 |
| 15583 | 192 | 269 | 331 |
| 15584 | 193 | 274 | 326 |
| 15585 | 213 | 315 | 353 |
| 15586 | 174 | 243 | 302 |
| 15587 | 181 | 260 | 324 |
| 15588 | 176 | 247 | 304 |
| 15589 | 171 | 247 | 284 |
| 15590 | 178 | 262 | 324 |
| 15591 | 158 | 230 | 279 |
| 15592 | 151 | 209 | 247 |
| 15593 | 170 | 238 | 260 |
| 15594 | 160 | 219 | 269 |
| 15595 | 201 | 279 | 295 |
| 15596 | 155 | 230 | 266 |
| Mean | 177.63 | 252.00 | 297.68 |
| Standard deviation | 16.72 | 25.71 | 29.04 |

Correlations

| | | | |
|---|---|---|---|
| $r_{IJ} = .968$ | $r_{IK} = .833$ | $r_{JK} = .871$ | $r_{EI} = .754$ |
| $r_{GI} = .775$ | $r_{GJ} = .802$ | $r_{GK} = .684$ | $r_{HK} = .665$ |

5% point = .456 1% point = .575

In Table III are given the results of diastatic activity determinations made upon samples of the same lots of wheat from which the flour samples were milled. These wheat samples were very finely ground in a steel burr mill (Seck) as described in the earlier section of this study. The agreement between wheat and flour diastatic activities in this series was quite good being $r_{GJ} = .80$ for the 60-minute digestion period, and a little lower for the other two periods. The agreement between digestion periods was not quite as good as in the case of the patent flours, the correlation varying from $r_{IK} = .83$ in the instance of 15 minutes versus 120 minutes digestion to $r_{IJ} = .97$ in the instance of 15 minutes versus 60 minutes digestion period.

The variability in the flour series was quite large, being at 60 minutes digestion from 94 to 246 Rumsey units in range. The larger part

of this variability can be assigned to that group previously described as varietal or inherent variations. The problem now was one to determine in what particular did these varieties differ in order to have shown so wide a spread in diastatic activity. The two major divisions in the diastatic activities of flours were shown by Mangels (1926) and Malloch (1929), as well as others since, to be enzyme concentration and starch susceptibility. The standard method of determining the concentration of the saccharifying enzymes has been the Lintner method in which the enzyme to be studied was allowed to react with soluble starch, the final amount of reducing sugars formed under fixed conditions being considered to be a measure of the enzyme concentration.

A modification of the Lintner method was developed which eliminated the necessity of separating an extract from the flour for the purpose of analysis since in the case of flours this subjects the determination to large errors and uncertainties. The method was to add 1 g. of Merck's soluble starch to the 5 g. of flour and conduct the determination upon the 6 g. of material in accordance with the directions of Blish and Sandstedt (1933). A blank determination was essential upon the soluble starch. A determination of diastatic activity of the flour alone should be run at the same time.

By such a procedure it should be possible to get a measure of the total amount of saccharifying enzymes present, since any enzymes which were unable to act upon the starch of the sample owing to its nature would be able to react with the added soluble starch. If an excess of sugar is produced over and above that produced from the starch of the flour samples, the Rumsey value would be a measure of starch susceptibility to diastatic attack rather than a measure of enzyme concentration.

This method was applied to the 19 flour samples described above. In all cases more sugar was formed when the soluble starch was present, indicating that the concentration of saccharifying enzymes was not the limiting factor in any case studied. The data are given in Table IV, in which the results are recorded as the amount of reducing sugars produced from 2 g. of soluble starch when the diastatic enzymes contained in 10 g. of flour acted simultaneously upon the soluble starch and the starch of the flour. This calculation was necessary in order to afford a direct comparison between the sugars produced from the soluble starch and the Rumsey figures for the flours. This method was used with the same four different digestion periods as before.

There was no consistent agreement between the diastatic activity of the flours as determined in the usual manner and the amount of sugar produced from the soluble starch by the same flours, the correlations ranging from $r_{FM} = -.32$ for the 30-minute digestion to $r_{GN} = +.58$ for the 60-minute period. The correlations between digestion periods

TABLE IV

AMOUNTS OF REDUCING SUGARS PRODUCED FROM TWO GRAMS MERCK'S SOLUBLE STARCH BY THE ACTION OF THE DIASTATIC ENZYMES CONTAINED IN TEN GRAMS OF PATENT FLOUR MILLED FROM 19 LOTS OF SPRING WHEAT OF THE 1933 CROP DURING VARYING DIGESTION PERIODS

(Milligrams)

| Flour sample number | 15 Min. *L* | 30 Min. *M* | 60 Min. *N* | 120 Min. *O* |
|---|---|---|---|---|
| 15578 | 52 | 50 | 58 | 64 |
| 15579 | 54 | 62 | 58 | 58 |
| 15580 | 73 | 69 | 62 | 58 |
| 15581 | 52 | 59 | 49 | 67 |
| 15582 | 55 | 62 | 63 | 65 |
| 15583 | 35 | 48 | 59 | 62 |
| 15584 | 52 | 60 | 55 | 63 |
| 15585 | 67 | 49 | 89 | 79 |
| 15586 | 70 | 57 | 65 | 60 |
| 15587 | 54 | 57 | 62 | 65 |
| 15588 | 64 | 60 | 60 | 80 |
| 15589 | 52 | 48 | 55 | 71 |
| 15590 | 69 | 70 | 58 | 80 |
| 15591 | 45 | 53 | 60 | 65 |
| 15592 | 58 | 61 | 63 | 66 |
| 15593 | 49 | 44 | 44 | 60 |
| 15594 | 50 | 68 | 51 | 68 |
| 15595 | 55 | 56 | 48 | 80 |
| 15596 | 55 | 57 | 71 | 76 |
| Mean | 55.84 | 57.37 | 59.47 | 67.74 |
| Standard deviation | 9.07 | 7.18 | 9.38 | 7.48 |

Correlations

$r_{LM} = .489$ $r_{LN} = .419$ $r_{LO} = .262$ $r_{MN} = -.048$ $r_{MO} = .054$

$r_{NO} = .254$ $r_{EL} = .165$ $r_{FM} = -.321$ $r_{GN} = .577$ $r_{HO} = .054$

5% point = .456 1% point = .575

for amount of soluble starch converted were also not high, none being statistically significant. Of the soluble starch converted to sugar in one hour's digestion 84% had been formed during the first 15 minutes, and 96% during the first 30 minutes, while prolonging the digestion period to 120 minutes increased the amount by 19%. Thus the rate of conversion of the soluble starch was very high at first but fell off even more rapidly than did the saccharification of starch of the flour samples. The results from this type of test are somewhat erratic owing to their being determined by difference, with four separate determinations entering into each value, so that even with each of the separate determinations in duplicate there are still many possibilities for cumulative errors.

This has shown that there was probably an abundance of the saccharifying or beta-amylase present in all the samples. This is in accord with the findings of Andrews and Bailey (1934). A limiting amount of alpha-amylase may have been the reason for only 2.6% of

TABLE V

AMOUNTS OF REDUCING SUGARS PRODUCED FROM VARYING AMOUNTS OF MERCK'S SOLUBLE STARCH BY THE ACTION OF THE DIASTATIC ENZYMES CONTAINED IN TEN GRAMS OF TWO SPRING WHEAT PATENT FLOURS DURING VARYING DIGESTION PERIODS

(Milligrams)

| Flour sample number | Weight of starch, grams | 15 Min. | 30 Min. | 60 Min. | 120 Min. |
|---|---|---|---|---|---|
| 15585 | 1.0 | 14 | 23 | 21 | 39 |
| | 2.0 | 67 | 49 | 89 | 79 |
| | 3.0 | 73 | 66 | 90 | 120 |
| 15588 | 1.0 | 34 | 34 | 34 | 34 |
| | 2.0 | 64 | 60 | 60 | 80 |
| | 3.0 | 97 | 86 | 108 | 123 |

the starch of the flour samples on the average being converted into reducing sugars in 120 minutes, but does not account for the fact that in the same time only 3.3% of the soluble starch was converted. Each of the flour samples might conceivably have had only a limited excess of beta-amylase, so in order to check this point two flours, one showing high and one low diastatic activity, were further studied, this time using 0.5 and 1.5 g. of the soluble starch superimposed upon the 5 g. of flour as well as the 1.0 g. and the flour alone. The results of these runs are given in Table V. It will be seen from these data that the amount of sugar formed from the solube starch was a function of the amount of soluble starch present and not a function of the concentration of alpha-amylase. This soluble starch was high quality and would readily form a water-clear solution.

TABLE VI

AMOUNTS OF REDUCING SUGARS FORMED FROM 10 GRAMS OF STARCH BY THE ACTION OF PURIFIED AMYLASES DURING VARYING DIGESTION PERIODS

| Kind of starch | Alpha-amylase | Beta-amylase | Digestion period | Reducing sugars |
|---|---|---|---|---|
| | *Cc.* | *Cc.* | *Min.* | *Mg.* |
| Merck's soluble | 5.0 | 5.0 | 30 | 240 |
| Dialysed corn | 0.0 | 0.0 | Blank | 0 |
| " " | 0.0 | 10.0 | 30 | 0 |
| " " | 0.0 | 0.0 | 30 | 0 |
| " " | 5.0 | 0.0 | 5 | 0 |
| " " | 5.0 | 0.0 | 30 | 0 |
| " " | 5.0 | 5.0 | 30 | 0 |

It appears probable from these studies that there is a third limiting factor in the enzymic conversion of starch to sugar, besides the enzyme and the starch. Just what this factor is is still uncertain, but a subsequent experiment throws a little light upon the subject. A sample of

well dialysed (84 hours) untreated corn-starch was subjected to hydrolysis by purified alpha- and beta-amylase, each singly, and also in combination without being able to produce any sugar. The combined enzymes would readily convert raw starches, and attacked the Merck's soluble starch at approximately the same rate as did the flour enzymes. The data of this experiment are given in Table VI. This factor is apparently associated with the starch since the extraction of the enzymes did not remove it, and the soluble starch retained it through the drastic chemical treatment, but upon prolonged dialysis it was either extracted from the starch or else was rendered inactive.

## Conclusions

Composite samples of patent flour appear to be as satisfactory for the determination of the relative diastatic power of different wheat varieties as the means of individual sample determinations; provided that only sound, ungerminated samples be used.

Wheat samples may be composited with nearly as great reliability as flour samples for diastatic activity determinations.

The digestion period in the diastatic activity determination upon flour may be shortened to 30 minutes without serious loss of accuracy.

An abundance of beta-amylase was found in all samples of flour studied.

A limiting factor for enzymatic saccharification of starch was found This lmiting factor was apparently associated with the starch and coulc only be removed or inactivated by prolonged dialysis.

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# FURTHER TESTS WITH MOTOR-DRIVEN DOUGH SHEETER

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(Read at the Annual Meeting, June, 1934)

In an effort to throw further light on the utility of the motor-driven dough sheeter for molding purposes in the American Association of Cereal Chemists' standard baking test, collaborative work was undertaken by The Fleischmann Laboratories, H. K. Parker and Robert Van Burek of the Wallace & Tiernan Laboratories, and Washington Platt and Ray Powers of the Borden Laboratories.

## Method and Apparatus

The same flours, and, as far as possible, the same apparatus was used in the three laboratories.

The method of study, except for mixing, was the same as that used last year at the Fleischmann Laboratories.[1] Each laboratory used a different type of mixer and oven, so that the mean volumes obtained in the different laboratories are not entirely comparable to each other, but, as will be pointed out below, the results obtained lead to some definite conclusions.

Two flours were used—a strong Northwestern patent, and a Southwestern patent.

Both sheeter and hand molding were used with each flour on a given day, and the same work was repeated on one or more subsequent days.

The same volume apparatus was used by all the laboratories.

Upon completion of the work in one laboratory, the volume apparatus and Thomson Dough Sheeter were shipped to the next laboratory.

It may be of interest to point out that the laboratories in which this work was done do not regularly use the "pup" loaf procedure, so that other laboratories unfamiliar with the test may be expected to get similar results if they attempt to duplicate our work.

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[1] Freilich, J. Molding tests with a motor-driven laboratory dough sheeter. Cereal Chem. 10: 585–588 (1933).

## Loaf Volumes and Statistical Analysis

The procedure followed at the Borden Laboratory with reference to the number of duplicates and the number of days, was somewhat different from that used by the Fleischmann and Wallace & Tiernan

TABLE I

LOAF VOLUMES OBTAINED BY THE FLEISCHMANN AND WALLACE & TIERNAN LABORATORIES WITH TWO FLOURS, WITH SHEETER AND HAND MOLDING, ON DIFFERENT DAYS

| Loaf | Fleischmann Laboratories | | Wallace & Tiernan Co. Laboratories | |
|---|---|---|---|---|
| | Sheeter | Hand | Sheeter | Hand |
| | *cc.* | *cc.* | *cc.* | *cc.* |
| | | *Flour No. 1, first day* | | |
| 1 | 564 | 595 | 560 | 545 |
| 2 | 543 | 580 | 562 | 535 |
| 3 | 553 | 565 | 563 | 567 |
| 4 | 552 | 595 | 567 | 630 |
| 5 | 565 | 575 | 562 | 605 |
| 6 | 574 | 585 | 582 | 585 |
| 7 | 567 | 599 | 568 | 615 |
| 8 | 573 | 611 | 570 | 610 |
| 9 | 573 | 603 | 585 | 605 |
| 10 | 570 | 604 | 605 | 620 |
| | | *Flour No. 2, first day* | | |
| 1 | 492 | 478 | 503 | 492 |
| 2 | 495 | 468 | 503 | 500 |
| 3 | 495 | 486 | 503 | 498 |
| 4 | 490 | 488 | 510 | 510 |
| 5 | 487 | 487 | 495 | 480 |
| 6 | 480 | 482 | 508 | 460 |
| 7 | 480 | 492 | 503 | 472 |
| 8 | 482 | 494 | 523 | 465 |
| 9 | 497 | 480 | 500 | 480 |
| 10 | 487 | 472 | 503 | 500 |
| | | *Flour No. 1, second day* | | |
| 1 | 550 | 593 | 612 | 570 |
| 2 | 560 | 587 | 585 | 597 |
| 3 | 558 | 592 | 615 | 600 |
| 4 | 566 | 600 | 575 | 595 |
| 5 | 555 | 585 | 627 | 615 |
| 6 | 538 | 597 | 590 | 585 |
| 7 | 555 | 608 | 595 | 590 |
| 8 | 553 | 610 | 590 | 590 |
| 9 | 550 | 616 | 597 | 570 |
| 10 | 556 | 615 | 595 | 575 |
| | | *Flour No. 2, second day* | | |
| 1 | 493 | 480 | 513 | 500 |
| 2 | 494 | 481 | 495 | 505 |
| 3 | 503 | 488 | 518 | 495 |
| 4 | 501 | 500 | 510 | 485 |
| 5 | 483 | 480 | 510 | 495 |
| 6 | 487 | 485 | 525 | 500 |
| 7 | 495 | 496 | 513 | 498 |
| 8 | 502 | 503 | 527 | 528 |
| 9 | 496 | 485 | 520 | 492 |
| 10 | 510 | 498 | 522 | 533 |

Laboratories; the Borden results will, therefore, be tabulated separately, and the Fleischmann and Wallace & Tiernan results will be given in the same tables.

Table I records the individual loaf volumes obtained at the Fleischmann and Wallace & Tiernan Laboratories, while Table II records the individual loaf volumes obtained at the Borden Laboratory.

TABLE II

LOAF VOLUMES OBTAINED BY THE BORDEN LABORATORY WITH TWO FLOURS, WITH SHEETER AND HAND MOLDING, ON DIFFERENT DAYS

| Loaf | Flour No. 1 Sheeter | Flour No. 1 Hand | Loaf | Flour No. 2 Sheeter | Flour No. 2 Hand |
|---|---|---|---|---|---|
| | *cc.* | *cc.* | | *cc.* | *cc.* |
| | | *First Day* | | | |
| 1 | 602 | 618 | 1 | 481 | 481 |
| 2 | 564 | 581 | 2 | 485 | 492 |
| 3 | 588 | 590 | 3 | 499 | 494 |
| 4 | 607 | 587 | 4 | 489 | 502 |
| 5 | 594 | 608 | 5 | 487 | 494 |
| | | *Second Day* | | | |
| 1 | 580 | 594 | 1 | 475 | 514 |
| 2 | 579 | 614 | 2 | 478 | 502 |
| 3 | 608 | 566 | 3 | 475 | 500 |
| 4 | 597 | 570 | 4 | 483 | 494 |
| 5 | 597 | 618 | 5 | 480 | 493 |
| | | *Third Day* | | | |
| 1 | 547 | 582 | 1 | 491 | 501 |
| 2 | 553 | 536 | 2 | 489 | 513 |
| 3 | 570 | 592 | 3 | 488 | 515 |
| 4 | 593 | 564 | 4 | 490 | 496 |
| 5 | 565 | 567 | 5 | 480 | 508 |
| | | *Fourth Day* | | | |
| 1 | 553 | 595 | 1 | 478 | 496 |
| 2 | 567 | 589 | 2 | 478 | 504 |
| 3 | 555 | 595 | 3 | 472 | 496 |
| 4 | 548 | 596 | 4 | 488 | 513 |
| 5 | 563 | 561 | 5 | 469 | 482 |

The volume figures in Tables I and II were subjected to statistical analysis, and the values for mean volume, standard deviation, and coefficient of variation are given in Tables III and IV.

A study of Tables III and IV shows the following;

(1) In fourteen out of sixteen comparisons between sheeter and hand molding, the sheeter gave smaller variations.

(2) There was no satisfactory agreement between the mean volumes obtained with the sheeter on different days in the same laboratory.

(3) There was no satisfactory agreement between the mean volumes obtained with the sheeter in the different laboratories; in fact, much better agreement was obtained by hand molding.

TABLE III

MEAN VOLUME, STANDARD DEVIATION AND COEFFICIENT OF VARIATION, CALCULATED FROM THE LOAF VOLUME FIGURES IN TABLE I

| | First day | | Second day | |
|---|---|---|---|---|
| | Sheeter | Hand | Sheeter | Hand |
| Flour No. 1, Fleischmann Laboratories | | | | |
| Mean volume, *cc.* | 563 | 591 | 554 | 600 |
| Standard deviation, *cc.* | 10.05 | 13.8 | 7.01 | 10.78 |
| Coefficient of variation, % | 1.78 | 2.33 | 1.27 | 1.79 |
| Flour No. 1, Wallace & Tiernan Laboratories | | | | |
| Mean volume, *cc.* | 572 | 592 | 598 | 589 |
| Standard deviation, *cc.* | 13.5 | 30.9 | 14.7 | 13.5 |
| Coefficient of variation, % | 2.36 | 5.22 | 2.46 | 2.29 |
| Flour No. 2, Fleischmann Laboratories | | | | |
| Mean volume, *cc.* | 489 | 483 | 496 | 490 |
| Standard deviation, *cc.* | 6.05 | 7.96 | 7.55 | 8.4 |
| Coefficient of variation, % | 1.24 | 1.65 | 1.52 | 1.71 |
| Flour No. 2, Wallace & Tiernan Laboratories | | | | |
| Mean volume, *cc.* | 505 | 486 | 515 | 503 |
| Standard deviation, *cc.* | 7.1 | 15.9 | 8.85 | 14.6 |
| Coefficient of variation, % | 1.41 | 3.26 | 1.72 | 2.9 |

TABLE IV

MEAN VOLUME, STANDARD DEVIATION AND COEFFICIENT OF VARIATION, CALCULATED FROM THE LOAF VOLUME FIGURES IN TABLE II

| | First day | | Second day | | Third day | | Fourth day | |
|---|---|---|---|---|---|---|---|---|
| | Sheeter | Hand | Sheeter | Hand | Sheeter | Hand | Sheeter | Hand |
| *Flour No. 1* | | | | | | | | |
| Mean volume, *cc.* | 591 | 597 | 592 | 592 | 566 | 568 | 557 | 587 |
| Standard deviation, *cc.* | 15 | 13.9 | 11.1 | 21.5 | 16 | 19 | 6.9 | 13.3 |
| Coefficient of variation, % | 2.54 | 2.33 | 1.88 | 3.62 | 2.83 | 3.34 | 1.24 | 2.26 |
| *Flour No. 2* | | | | | | | | |
| Mean volume, *cc.* | 488 | 493 | 478 | 501 | 488 | 507 | 477 | 498 |
| Standard deviation, *cc.* | 6 | 7 | 3.1 | 7.55 | 3.9 | 7.2 | 6.5 | 10.2 |
| Coefficient of variation, % | 1.23 | 1.42 | 0.65 | 1.5 | 0.8 | 1.42 | 1.36 | 2.04 |

The last point may be brought out more clearly, as shown in Tables V and VI, by placing the mean volumes obtained in the different laboratories side by side, and then comparing their variations from the mean.

TABLE V

COMPARISON OF MEAN VOLUMES OBTAINED IN THE THREE LABORATORIES BY SHEETER AS AGAINST HAND MOLDING Figures from Tables III and IV

| Laboratory | Day | Sheeter | Hand | Sheeter | Hand |
|---|---|---|---|---|---|
| | | *cc.* | *cc.* | *cc.* | *cc.* |
| | | *Flour No. 1* | | *Flour No. 2* | |
| Fleischmann | 1st | 563 | 591 | 489 | 483 |
| | 2nd | 554 | 600 | 496 | 490 |
| Wallace & Tiernan | 1st | 572 | 592 | 505 | 486 |
| | 2nd | 598 | 589 | 515 | 503 |
| Borden | 1st | 591 | 597 | 488 | 493 |
| | 2nd | 592 | 592 | 478 | 501 |
| | 3rd | 566 | 568 | 488 | 507 |
| | 4th | 557 | 587 | 477 | 498 |

TABLE VI

MEAN VOLUME, STANDARD DEVIATION, AND COEFFICIENT OF VARIATION CALCULATED FROM THE LOAF VOLUME Figures in Table V

| Statistic | Sheeter | Hand | Sheeter | Hand |
|---|---|---|---|---|
| | *Flour No. 1* | | *Flour No. 2* | |
| Mean volume, *cc.* | 574 | 590 | 492 | 495 |
| Standard deviation, *cc.* | 16.05 | 9.02 | 12.1 | 7.94 |
| Coefficient of variation, % | 2.8 | 1.53 | 2.46 | 1.9 |

The mean volume, standard deviation, and coefficient of variation, calculated from the figures in Table V are given in Table VI.

Table VI shows that, when comparing the mean volumes obtained in the different laboratories, the variations with hand molding were much smaller than those obtained with the sheeter, for both flours.

## Summary

The writer believes that a satisfactory standard molding procedure should meet the following specifications:

Give reproducible results with a minimum of variation.

Require a minimum number of duplicate bakings.

Give reproducible results at different times in the same laboratory.

Give reproducible results when used in different laboratories.

The last is perhaps the most important requirement if the main purpose is really to have a method which will give approximately the same results when used in different laboratories, because no matter how well the other requirements are satisfied, the method will not be standard, in the best sense of the word, if fourth item above is not satisfied.

It is, therefore, concluded that the sheeter will not serve our purpose, because, as shown by these results, it does not meet the requirements of a satisfactory standard molding procedure.

This conclusion—that the sheeter is unsatisfactory—is valid only if we agree to accept the four requirements arbitrarily listed above. From this viewpoint, hand molding is, of course, also unsatisfactory. Each method has its own advantages. The sheeter is decidedly to be preferred over hand molding if one wishes to obtain better results when comparing different samples on a given day in his own laboratory. Hand molding, on the other hand, is to be preferred over the sheeter when better agreement between different laboratories is desired.

That hand molding would give better agreement between laboratories, even though the sheeter gave best results in any one laboratory, was not expected. A possible explanation of this is the assumption that the greater number of errors in all directions involved in hand molding, produced a statistical average in which these errors neutralized each other more so than in the averages for sheeter molding, where the errors in one laboratory may have gone in one direction, while those in another laboratory went in an opposite direction, due to that part of the molding which was done by hand.

But, whatever the explanation may be, the encouraging results obtained by the use of the sheeter in eliminating some of the daily errors involved in the hand-molding procedure, lead to the belief that a completely mechanized procedure would prove more satisfactory, for all purposes, than either sheeter or hand molding.

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## MIXING-TIME EXPERIMENTS WITH FLOURS MILLED FROM DIFFERENT CLASSES OF WHEATS

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(Read at the Annual Meeting, June 1934)

In three previous reports (Heald (1930), Merritt and Blish (1931), and Merritt, Blish, and Sandstedt (1932)), are recorded a series of mixing studies using the Hobart-Swanson mixer. The superiority of this new dough mixer, when compared with those previously used in experimental baking laboratories, cannot be questioned. Other mixing studies reveal (Swanson and Working, 1933) that different flours when mixed in the Hobart-Swanson machine do not all behave in the same or

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similar manner. The weaker flours require a shorter mixing time as compared with the stronger flours. In some instances the stronger flours may be mixed up to seven minutes without showing any marked breaking down or injury as measured in terms of the baked loaf.

In the final report of the A.A.C.C. Baking Fellow, (Merritt, Blish, and Sandstedt, 1932) the recommendation of one minute mixing period was made as sufficient to facilitate an even distribution of the ingredients without developing the gluten.

The following studies reported in this paper were made to establish a mixing time best suited to the flours milled from the different commercial classes of wheat and to the various grades of flour.

For the purpose of this study a number of experimentally milled flours as well as three commercially milled flours were selected. The baking data are recorded in Tables I, II, III, IV. In Table I are listed the baking data obtained with the experimentally milled soft white wheat flours; Table II the baking data obtained with the soft red winter flours; Table III the baking data for the hard red winter and hard red spring flours; while in Table IV are to be found the data obtained with commercial flours milled from hard red spring wheats.

In these tests 200 grams of flour was doughed and divided into equally weighed portions immediately after mixing. Eight replicate bakes were made. The weighed average of the eight bakes is given in connection with the various mixing times. The mixing times used were ½, ¾, 1, 1½, 2 and 3 minutes.

The A.A.C.C. standard basic procedure was used only in the instance of the commercially milled flours as it was found unsuited for the experimentally milled samples. Experimentally milled flours require more sugar and a shorter fermentation period. The proofing and baking times recommended for the standard basic procedure were followed however. The different formulas used are given in the tables along with the baking and chemical characteristics of the flours.

With the soft white flours (Table I) there was little change in loaf volume with mixing times up to 1½ minutes. The exception to this is the sample of flour milled from Federation wheat which showed a marked increase in loaf volume when mixed for one minute.

With these white wheat flours extended mixing for two and three minutes produced a marked improvement in the internal structure of the loaf in every instance. Increases in loaf volume, however, were marked only in the instance of the flours milled from the varieties Federation, Baart, and Pacific Bluestem.

The mixing data obtained with the soft red winter wheat flours are shown in Table II. The bread scores show an improvement as the time of mixing is extended with the loaf volumes remaining practically

TABLE I

STUDIES WITH THE HOBART-SWANSON MIXER. EFFECT ON BREAD CHARACTERISTICS BY VARYING THE DOUGH MIXING TIME FROM ½ TO 3 MINUTES

*Experimentally milled soft white wheat flours*

| Type of flour | Length of mixing | Loaf volume | Variation in loaf volume between duplicate determinations | Loaf volume deviation from ½ minute mixing | | Color of crumb | Deviation of color of crumb from ½ minute mixing | Grain and texture | Deviation of grain and texture from ½ minute mixing | Color of crust | Protein[1] | Ash[1] | Dough characteristics |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | *Minutes* | *Cc.* | % | *Cc.* | % | | | | | | % | % | |
| Michigan White | ½ | 472 | 2.8 | — | — | 70 Cr. | — | 65 F. | — | S.B. | 8.84 | 0.44 | Mixed smooth |
| | ¾ | 471 | 2.4 | − 1 | − .21 | 70 Cr. | — | 65 F. | — | " | — | — | " " |
| | 1 | 472 | .9 | 0 | 0 | 70 Cr. | — | 65 F. | — | " | — | — | " " |
| | 1½ | 477 | 1.5 | +5 | +1.06 | 75 Cr. | + 5 | 70 F. | + 5 | " | — | — | " " |
| | 2 | 479 | 1.9 | +7 | +1.42 | 80 Cr. | +10 | 70 F. | + 5 | L.B. | — | — | Broke down in mixing, sticky |
| | 3 | 484 | 1.8 | +12 | +2.44 | 85 Cr. | +15 | 75 F. | +10 | " | — | — | Broke down in mixing, very sticky |
| Pacific Coast Federation | ½ | 502 | .9 | — | — | 75 Cr. | — | 70 F. | — | S.B. | 8.57 | 0.45 | Undermixed |
| | ¾ | 531 | 2.2 | +29 | +5.78 | 75 Cr. | — | 75 F. | + 5 | " | — | — | Mixed smooth |
| | 1 | 538 | 1.8 | +36 | +7.17 | 80 Cr. | + 5 | 80 F. | +10 | B. | — | — | " " |
| | 1½ | 531 | 2.8 | +29 | +5.78 | 80 Cr. | + 5 | 85 F. | +15 | " | — | — | " " |
| | 2 | 539 | 1.4 | +37 | +7.37 | 85 Cr. | +10 | 85 F. | +15 | " | — | — | " " |
| | 3 | 542 | 1.3 | +40 | +7.97 | 90 Cr. | +15 | 90 G. | +20 | " | — | — | Broke down in mixing, sticky |
| Pacific Coast soft white | ½ | 524 | .9 | — | — | 75 Cr. | — | 75 F. | — | S.B. | 9.21 | 0.44 | Undermixed |
| | ¾ | 534 | 1.6 | +10 | +1.91 | 75 Cr. | — | 75 F. | — | B. | — | — | Mixed smooth |
| | 1 | 535 | 1.8 | +11 | +2.10 | 75 Cr. | — | 75 F. | — | " | — | — | " " |
| | 1½ | 529 | 1.4 | + 5 | + .95 | 80 Cr. | + 5 | 75 F. | — | " | — | — | " " |
| | 2 | 542 | 1.8 | +18 | +3.44 | 80 Cr. | + 5 | 80 F. | + 5 | " | — | — | " " |
| | 3 | 543 | 1.1 | +19 | +3.63 | 85 Cr. | +10 | 80 F. | + 5 | " | — | — | Broke down in mixing, sticky |
| Pacific Coast Club | ½ | 512 | 1.5 | — | — | 70 Cr. | — | 70 F. | — | L.B. | 8.02 | 0.39 | Undermixed |
| | ¾ | 513 | 1.2 | + 1 | + .20 | 70 Cr. | — | 70 F. | — | " | — | — | Mixed smooth |
| | 1 | 502 | 1.9 | −10 | −1.95 | 75 Cr. | + 5 | 75 F. | + 5 | " | — | — | " " |
| | 1½ | 507 | 1.7 | − 5 | − .98 | 75 Cr. | + 5 | 75 F. | + 5 | " | — | — | " " |
| | 2 | 517 | 2.7 | + 5 | + .98 | 80 Cr. | +10 | 80 F. | +10 | " | — | — | " " |
| | 3 | 515 | 1.2 | + 3 | + .59 | 85 Cr. | +15 | 85 F. | +15 | S.B. | — | — | Broke down in mixing, sticky |
| Pacific Coast Bluestem | ½ | 494 | 4.1 | — | — | 60 Cr. gr. | — | 60 P. | — | B. | 11.77 | 0.53 | Undermixed, slightly stiff |
| | ¾ | 508 | .9 | +14 | +2.83 | 60 Cr. gr. | — | 60 P. | — | " | — | — | Mixed smooth, " " |
| | 1 | 507 | 2.3 | +13 | +2.63 | 60 Cr. gr. | — | 60 P. | — | " | — | — | " " " " |
| | 1½ | 515 | .9 | +21 | +4.25 | 65 Cr. gr. | + 5 | 65 F. | + 5 | " | — | — | " " " " |
| | 2 | 554 | .8 | +60 | +12.15 | 75 Cr. gr. | +15 | 75 G. | +15 | " | — | — | " " " " |
| | 3 | 596 | 1.3 | +102 | +20.65 | 80 Cr. gr. | +20 | 70 F. | +10 | " | — | — | Broke down in mixing, sticky |

[1] Calculated to 13.5 percent moisture basis.

Formula used:
5 % sugar
1½ % salt
2 % yeast
54 % absorption

2½ hrs. fermentation
First punch 80 min.
Second punch 45 "
Pan 25 "

## TABLE II

STUDIES WITH THE HOBART-SWANSON MIXER. EFFECT ON BREAD CHARACTERISTICS BY VARYING THE DOUGH MIXING TIME FROM ½ TO 3 MINUTES

*Experimentally milled soft red winter wheat flours.*

| Type of flour | Length of mixing | Loaf volume | Variation in loaf volume between duplicate determinations | Loaf volume deviation from ½ minute mixing | | Color of crumb | Deviation of color of crumb from ½ minute mixing | Grain and texture | Deviation of grain and texture from ½ minute mixing | Color of crust | Protein[1] | Ash[1] | Dough characteristics |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | *Minutes* | *Cc.* | % | *Cc.* | % | | | | | | % | % | |
| Maryland soft red winter | ½ | 523 | 1.3 | — | — | 70 Cr. gr. | — | 65 F. | — | L.B. | 8.78 | 0.46 | Undermixed |
| | ¾ | 526 | 1.5 | + 3 | + .57 | 70 Cr. gr. | — | 70 F. | + 5 | B. | — | — | Mixed smooth |
| | 1 | 528 | 1.4 | + 5 | + .96 | 70 Cr. gr. | — | 75 F. | +10 | " | — | — | " " |
| | 1½ | 522 | 1.9 | − 1 | − .19 | 75 Cr. gr. | + 5 | 75 F. | +10 | " | — | — | " " |
| | 2 | 538 | 1.3 | +15 | +2.87 | 75 Cr. gr. | + 5 | 75 F. | +10 | " | — | — | " " |
| | 3 | 528 | 1.2 | + 5 | + .96 | 80 Cr. gr. | +10 | 75 F. | +10 | " | — | — | Broke down in mixing, sticky |
| Maryland soft red winter | ½ | 523 | 2.6 | — | — | 65 Cr. gr. | — | 65 F. | — | L.B. | 8.81 | 0.50 | Undermixed |
| | ¾ | 528 | 1.7 | + 5 | + .96 | 65 Cr. gr. | — | 70 F. | + 5 | B. | — | — | Mixed smooth |
| | 1 | 530 | 1.7 | + 7 | +1.34 | 65 Cr. gr. | — | 75 F. | +10 | " | — | — | " " |
| | 1½ | 528 | 1.9 | + 5 | + .96 | 70 Cr. gr. | + 5 | 75 F. | +10 | " | — | — | " " |
| | 2 | 533 | 1.1 | +10 | +1.91 | 70 Cr. gr. | + 5 | 75 F. | +10 | " | — | — | " " |
| | 3 | 528 | 1.1 | + 5 | + .96 | 75 Cr. gr. | +10 | 75 F. | +10 | " | — | — | Broke down in mixing, sticky |

[1] Calculated to 13.5 percent moisture basis.

Formula used:
5 % sugar
1½ % salt
2 % yeast
54 % absorption

3 hrs. fermentation
First punch 105 min.
Second punch 50 "
Pan 25 "

## TABLE III

STUDIES WITH THE HOBART-SWANSON MIXER. EFFECT ON BREAD CHARACTERISTICS BY VARYING THE DOUGH MIXING TIME FROM ½ TO 3 MINUTES

*Experimentally milled hard red winter and hard red spring wheat flours*

| Type of flour | Length of mixing | Loaf volume | Variation in loaf volume between duplicate determinations | Loaf volume deviation from ½ minute mixing | | Color of crumb | Deviation of color of crumb from ½ minute mixing | Grain and texture | Deviation of grain and texture from ½ minute mixing | Color of crust | Protein[1] | Ash[1] | Dough characteristics |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | *Minutes* | *Cc.* | % | *Cc.* | % | | | | | | % | % | |
| Hard red winter | ½ | 576 | 2.8 | — | — | 100 Cr. | — | 95 G. | — | B. | 10.74 | 0.45 | Undermixed |
| | ¾ | 571 | .5 | − 5 | − .87 | 100 Cr. | — | 100 G. | + 5 | " | — | — | " |
| | 1 | 577 | 1.5 | + 1 | + .17 | 100 Cr. | — | 105 G. | +10 | " | — | — | Mixed smooth |
| | 1½ | 580 | 2.1 | + 4 | + .69 | 105 Cr. | + 5 | 110 V.G. | +15 | " | — | — | " " |
| | 2 | 592 | 2.5 | +16 | +2.78 | 105 Cr. | + 5 | 110 V.G. | +15 | " | — | — | " " |
| | 3 | 601 | 1.0 | +25 | +4.34 | 110 Cr. | +10 | 110 V.G. | +15 | " | — | — | " " sticky |
| Hard red spring | ½ | 569 | 1.3 | — | — | 90 Cr. | — | 80 G. | — | B. | 12.82 | 0.48 | Undermixed |
| | ¾ | 579 | 2.1 | +10 | +1.76 | 90 Cr. | — | 80 G. | — | " | — | — | " |
| | 1 | 585 | 2.3 | +16 | +2.81 | 90 Cr. | — | 85 G. | + 5 | " | — | — | Mixed smooth |
| | 1½ | 607 | 2.0 | +38 | +6.68 | 95 Cr. | + 5 | 90 V.G. | +10 | " | — | — | " " |
| | 2 | 643 | 1.8 | +74 | +13.01 | 100 Cr. | +10 | 95 V.G. | +15 | " | — | — | " " |
| | 3 | 667 | 2.1 | +98 | +17.22 | 105 Cr. | +15 | 100 V.G. | +20 | " | — | — | " " |

[1] Calculated to 13.5 percent moisture basis.

Formula used for hard red winter:

| | | | |
|---|---|---|---|
| 5 % Sugar | 3 hrs. fermentation | | |
| 1½ % Salt | First punch | 105 min. | |
| 2 % Yeast | Second punch | 50 " | |
| 58 % Absorption | Pan | 25 " | |

Formula used for hard red spring:

| | | | |
|---|---|---|---|
| 3½ % Sugar | 2 hrs. 40 min. fermentation | | |
| 1½ % Salt | First punch | 90 min. | |
| 3 % Yeast | Second punch | 45 " | |
| 60 % Absorption | Pan | 25 " | |

## TABLE IV

STUDIES WITH THE HOBART-SWANSON MIXER. EFFECT ON BREAD CHARACTERISTICS BY VARYING THE DOUGH MIXING TIME FROM ½ TO 3 MINUTES

*Commercially milled hard red spring wheat flours*

| Type of flour | Length of mixing | Loaf volume | Variation in loaf volume between duplicate determinations | Loaf volume deviation from ½ minute mixing | | Color of crumb | Deviation of color of crumb from ½ minute mixing | Grain and texture | Deviation of grain and texture from ½ minute mixing | Color of crust | Protein[1] | Ash[1] | Dough characteristics |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | *Minutes* | *Cc.* | % | *Cc.* | % | | | | | | % | % | |
| Bakers standard patent | ½ | 672 | .8 | — | — | 95 Cr. | — | 85 G. | — | B. | 14.77 | 0.50 | Undermixed |
| | ¾ | 683 | 1.7 | +11 | +1.64 | 95 Cr. | — | 90 V.G. | + 5 | " | — | — | " |
| | 1 | 698 | 2.4 | +26 | +3.87 | 95 Cr. | — | 95 V.G. | +10 | " | — | — | Mixed smooth |
| | 1½ | 731 | .8 | +59 | +8.78 | 100 Cr. | + 5 | 95 V.G. | +10 | " | — | — | " " |
| | 2 | 737 | 1.6 | +65 | +9.67 | 105 Cr. | +10 | 105 V.G. | +20 | " | — | — | " " |
| | 3 | 761 | 1.5 | +89 | +13.24 | 105 Cr. | +10 | 105 V.G. | +20 | " | — | — | " " tough sticky |
| Bakers standard patent | ½ | 610 | 1.0 | — | — | 100 Cr. | — | 80 G. | — | B. | 13.05 | 0.50 | Undermixed, blistered on top at first punch |
| | ¾ | 619 | 3.5 | + 9 | +1.48 | 95 Cr. | − 5 | 80 G. | — | " | — | — | Undermixed, blistered on top at first punch |
| | 1 | 637 | 2.2 | +27 | +4.43 | 100 Cr. | — | 85 V.G. | + 5 | " | — | — | Mixed smooth |
| | 1½ | 657 | 1.4 | +47 | +7.70 | 105 Cr. | + 5 | 90 V.G. | +10 | " | — | — | " " |
| | 2 | 677 | .8 | +67 | +10.98 | 105 Cr. | + 5 | 90 V.G. | +10 | " | — | — | " " |
| | 3 | 669 | 1.0 | +59 | +9.67 | 105 Cr. | + 5 | 90 V.G. | +10 | " | — | — | " " slightly sticky |
| First clear | ½ | 576 | .5 | — | — | 65 Cr. gr. | — | 75 F. | — | B. | 14.14 | 0.78 | Undermixed, blistered on top at first punch |
| | ¾ | 566 | 2.8 | −10 | −1.74 | 65 Cr. gr. | — | 75 G. | — | " | — | — | Undermixed, blistered on top at first punch |
| | 1 | 566 | 2.5 | −10 | −1.74 | 65 Cr. gr. | — | 80 G. | + 5 | " | — | — | Mixed smooth, blistered on top at first punch |
| | 1½ | 576 | 2.8 | 0 | 0 | 65 Cr. gr. | — | 80 G. | + 5 | " | — | — | Mixed smooth, blistered on top at first punch |
| | 2 | 592 | 1.0 | +16 | +2.78 | 70 Cr. gr. | + 5 | 85 G. | +10 | " | — | — | Mixed smooth, blistered on top at first punch |
| | 3 | 597 | 1.7 | +21 | +3.65 | 75 Cr. gr. | +10 | 90 G. | +15 | " | — | — | Mixed smooth, blistered on top at first punch |

[1] Calculated to 13.5 percent moisture basis.

A.A.C.C. formula used:
2½% Sugar
1 % Salt
3 % Yeast
65 % Absorption

3 hrs. fermentation
First punch 105 min.
Second punch 50 "
Pan 25 "

the same over the entire mixing time range of three minutes. It will be observed, by referring to the data recorded in Tables I and II, that experimentally milled soft white and soft red winter wheat flours are (1) undermixed at ½ minute, (2) that ¾ minute mixing is the minimum time at which the ingredients are all incorporated to form a smooth dough, (3) that at 1 and again at 1½ minutes of mixing better internal loaf characteristics are evident, (4) that the gluten is apparently slightly developed at 1, and also 1½ minutes, and finally, (5) the doughs become sticky and plastic after 2 and 3 minutes mixing. It is recommended, therefore, that a ¾ minute mixing time is optimum for experimentally milled soft white and soft red winter wheat flours.

The data given in Table III, for the experimentally milled hard red spring and hard red winter wheat flours, reveal marked betterment in grain and texture and color of crumb scores as the mixing time is increased. The loaf volumes obtained with the flours milled from hard red winter wheats showed less increase over the mixing range than did the hard red spring flours. Loaves from flours milled from both of these types of wheat had the appearance of being undermixed at ½ and ¾ minute intervals. The one minute mix produced a smooth dough with both samples, and only in the event of the hard red winter flour did the dough evidence signs of breaking down with extended mixing. It was obvious that a minute or even a 1½ minutes mixing time can be recommended for this type of hard wheat flour.

The baking data for the commercially milled hard red spring wheat flours are shown in Table IV. Three grades of flour were used. With these flours the doughs were undermixed at ½ and ¾ minutes but produced a smooth dough at one minute. It is also evident that while the improvement in color of crumb was nominal, there was a marked improvement in grain and texture due to extended mixing with all three grades of flour.

The sample of first clear flour did not respond to mixing as is shown by the loaf volumes, which are practically the same for the range in mixing times.

It is recommended, therefore, that a 1 to 1½ minutes mix with the Hobart-Swanson mixer is sufficient for commercially milled hard red spring flour samples.

The factors contributing to the variability in loaf volume between duplicate tests have always been a question among experimental workers since the baking test originated. Numerous laboratory tests have been performed under controlled conditions in an effort to obtain duplicate results which would approach the accuracy of the chemical determinations made on wheat and flour. Inspection of the data presented for the various flour samples under the columns headed

"Variation in Loaf Volume Between Duplicate Determinations" evidenced a wide range of variability.

In summarizing these data the average variation for the different mixing times is as follows:

| | |
|---|---|
| ½ minute | 1.7% |
| ¾ minute | 1.7% |
| 1 minute | 1.8% |
| 1½ minutes | 1.8% |
| 2 minutes | 1.5% |
| 3 minutes | 1.3% |

It will be noted from these average figures that as the time of mixing is extended that the variability between duplicate tests becomes less. The experiments have not thrown any light on the casual factors involved, but it is suspected that gluten development and a thorough incorporation of the ingredients are responsible for greater uniformity between duplicate tests in the instances of the longer mixed samples.

## Conclusions

A number of experimentally and commercially milled flours were mixed for varying lengths of time from ½ minute to 3 minutes in the Hobart-Swanson mixer.

It appears from these studies that:

¾ of a minute mixing is sufficient to incorporate the ingredients for the experimentally milled soft red winter and soft white wheat flours.

1 to 1½ minutes mixing is sufficient to incorporate the ingredients for the experimentally and commercially milled hard red spring wheat flours, and the experimentally milled hard red winter wheat flours.

The mixing times as recommended for the different classes of wheat flours are the minimum limit at which there is no apparent gluten development.

Prolonged mixing effecting an increase in loaf volume, improved grain and texture, and lighter crumb color, is seemingly a result of gluten development.

Extended mixing intervals of 2 and 3 minutes results in less variability between duplicate tests.

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# A MODIFIED METHOD FOR THE REMOVAL OF ADDED INGREDIENTS FROM PHOSPHATED AND SELF-RISING FLOURS IN ORDER TO DETERMINE THE ASH CONTENT OF THE ORIGINAL FLOUR [1, 2]

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(Received for publication February 18, 1935)

## Introduction

It is generally agreed that the ash content of flour, expressed in percentage on the basis of 15% moisture, is the most reliable and most widely used criterion of flour grades for commercial purposes. The addition of inorganic salts as flour improvers and leavening agents, such as those used in phosphated and self-rising flours, considerably increases the total ash content of such products. The determination of the ash content of such flours without first removing the added ingredients therefore does not indicate the true grade of the original flour.

Natural mineral matter in flour is intimately associated with other flour components, and is not easily separated therefrom when brought into contact with certain non-aqueous liquids. Although it has been known that carbon tetrachloride is a suitable reagent for the removal of added phosphate from commercial wheat flours, only the procedure of Gustafson [3] has come into use. He has shown that this reagent can be used to effect a removal of added phosphate and other commonly used self-rising ingredients from flour. A quantitative determination of the ash content of the original flour is thus afforded. His method calls for a 20-gram charge of flour in a metal centrifuge tube cup, 2 inches in diameter and 6 inches deep, to which about 250 cc. of carbon tetrachloride is added and then centrifuged from 5 to 7 minutes at a speed of 1600 r.p.m. The flour is skimmed off, allowed to dry overnight, and analyzed for moisture and ash as usual.

Considerable experience with the above method indicated that certain conditions of the method should receive further study. It is highly de-

[1] Published with permission of the Director of the University of Tennessee Agricultural Experiment Station.

[2] Acknowledgment is made to the J. Allen Smith & Company, who have provided materials for this work.

[3] Cereal Chemistry, Vol. VIII, No. 6, November, 1931.

sirable that (*a*) the flour sample be intimately mixed with the carbon tetrachloride, especially when lumpy, or "shot-ball," flour is encountered; (*b*) that the surface layer of flour be prevented from drying during centrifuging; and (*c*) that the method be made more economical as to time and material. As a result of considerable preliminary experimental work toward these objectives, the following modified method was developed.

## Method

The sample should be thoroughly mixed by *rolling* diagonally across paper, alternating the direction of rolling some 25 to 30 times. Weigh a 20-gram charge of the well-mixed flour and transfer into a clean, dry Squibb separatory funnel [4] of 150 cc. capacity. Add 50 cc. of pure carbon tetrachloride; close the funnel with a short cork and *shake vigorously* and continuously for one minute. This is very important to disintegrate lumps and to insure intimate contact of the liquid and flour particles. Place the funnel in a Trunnion carrier [5] specially designed for the Squibb funnel, making sure that the funnel is firmly seated in the lower rubber cushion; and without removing the cork, centrifuge the suspension at a speed of between 1000 and 1100 r.p.m. for seven minutes. Permit the centrifuge to come to rest gradually. Carefully remove the cork; open the stopcock, and allow the sediment together with about 30 to 35 cc. of the liquid to run out, retaining about 10 to 12 cc. in the funnel. (In most cases the sediment and liquid will flow out readily. Should this not happen, puncture the flour layer with a thin glass rod drawn to a point and slightly rounded at the end. The rod may also be used to dislodge traces of sediment that may occasionally adhere to the funnel just above the stopcock.) Introduce 25 cc. of carbon tetrachloride, shake for 30 seconds, stopper, and centrifuge for 5 minutes. Remove the cork and run through the stopcock all but 5 to 7 cc. of the liquid. Close the funnel and shake until the flour is thinned by the remaining liquid, and pour into a wide, shallow glass dish (Petri dishes size 100 × 10 mm. serve well for this purpose). Dry the flour by placing the dish about 12 inches from an electric heating fan. At a temperature of about 45° C. in a current of air, the odor of carbon tetrachloride will be completely dispelled in from 20 to 30 minutes. The dried flour is then pulverized and mixed, and is ready for the determinations of moisture and ash. The total time required to prepare samples as above described is about one hour.

## Experimental

The efficiency of the method was tested by using plain soft wheat flour as a control. Four definite mixtures were prepared: (*a*) a mixture of control flour and sodium bicarbonate; (*b*) one of control flour and sodium chloride; (*c*) one of control flour and mono-calcium phosphate; and (*d*) one of control flour and the three salts. The soda, salt, and phosphate used were high-grade commercial products. Charges of 20 g. of each mixture were treated with carbon tetrachloride according to the above method, and analyzed as usual for moisture and for ash. The results are recorded in Table I, on the basis of 15% moisture.

As shown in Table I, concordant ash results were obtained on flours from which the several added salts were removed; and all ash results agree very closely with that of the control flour, the maximum difference being 0.0025%.

[4] Stopcocks of separatory funnels should be lubricated with a good grade of grease such as "Cello-grease." Vaseline will not hold the stopcocks in place. As a secondary precaution, a wide rubber band may be wound around funnel and stopcock before placing in holder.

[5] Special Trunnion carriers for use with the Squibb separatory funnel may be purchased from the International Centrifuge Co.

TABLE I

ASH CONTENT OF PLAIN SOFT WHEAT FLOUR BEFORE AND AFTER REMOVAL OF ADDED SALTS WITH CARBON TETRACHLORIDE

| Flour sample | Ash content of flours [1] | | |
|---|---|---|---|
| | Before removal of added salts | After removal of added salts | Deviation from control flour |
| | % | % | % |
| Control | 0.415 | | |
| | .413 | | |
| | Ave. .414 | —— | —— |
| Control treated with carbon tetrachloride | .418 | | |
| | .414 | | |
| | Ave. .416 | —— | +.002 |
| Control and soda | 1.096 | 0.4130 | |
| | | .4100 | |
| | | Ave. .4115 | −.0025 |
| Control and salt | 2.404 | .4150 | |
| | | .4100 | |
| | | Ave. .4125 | −.0015 |
| Control and phosphate | 1.659 | .4160 | |
| | | .4130 | |
| | | Ave. .4145 | +.0005 |
| Control, soda, salt, and phosphate | 3.935 | .4160 | |
| | | .4140 | |
| | | Ave. .4150 | +.001 |

| | |
|---|---|
| Average difference in ash of control flour and ash of same flour after removing added salts | 0.0015 |
| Maximal difference | 0.0025 |
| Minimal difference | 0.0005 |

[1] Results on the basis of 15% moisture.

It is important to impose uniform conditions in carrying out the modified procedure. This was shown as follows: Charges of 20 g. of self-rising flour, prepared by mixing together definite weights of control flour, soda, salt, and phosphate, were treated with varying amounts of carbon tetrachloride, and centrifuged at a speed of about 1010 r.p.m. for different periods, other conditions remaining constant. Moisture and ash were determined on the recovered flour. The results obtained under the imposed conditions are shown in Table II.

The data of Table II demonstrate that accurate results can be obtained under conditions prescribed in the modified method, and that inaccuracies occur when conditions are not controlled. The ash results of trials 1, 2, and 3, are in close agreement. Although apparently unnecessary, centrifuging for 10 minutes does no harm. When less than 50 cc. of carbon tetrachloride is used, the salts are incompletely removed,

as will be noted by inspection of data for trials 4 and 5. A second treatment of sample with carbon tetrachloride is apparently necessary to insure the complete removal of self-rising ingredients. High ash contents were registered by trials 6 and 7 which received a single treatment with an increased amount of liquid. The ash of trial 8, that received the second treatment, agrees very closely with the ash of the control flour. An adequate centrifuging period is essential. This is shown by trials 9 and 10, where the period of centrifuging was reduced from 7 to 5 minutes for the first treatment, and from 5 to 3 minutes for the second, the ash results of which are high. Trials 11 and 12 received 250 cc. of carbon tetrachloride, and were centrifuged for 7 and 10 minutes, respectively, and gave high ash results.

TABLE II

ASH CONTENT OF FLOUR RECOVERED FROM SELF-RISING FLOUR UNDER VARYING CONDITIONS

| Trial number | First Treatment | | Second Treatment | | Ash content of recovered flour [1] | Deviation from control flour [2] |
|---|---|---|---|---|---|---|
| | Volume of carbon tetrachloride | Period of centrifuging | Volume of carbon tetrachloride | Period of centrifuging | | |
| | *Cc.* | *Min.* | *Cc.* | *Min.* | % | % |
| 1 | 50 | 7 | 25 | 5 | 0.415 | +.001 |
| 2 | 50 | 7 | 25 | 5 | .419 | +.005 |
| 3 | 50 | 10 | 25 | 5 | .415 | +.001 |
| 4 | 25 | 7 | 25 | 5 | .425 | +.011 |
| 5 | 25 | 10 | 25 | 5 | .428 | +.014 |
| 6 | 75 | 7 | — | — | .439 | +.025 |
| 7 | 75 | 10 | — | — | .455 | +.041 |
| 8 | 75 | 7 | 25 | 5 | .415 | +.001 |
| 9 | 50 | 5 | 25 | 3 | .424 | +.010 |
| 10 | 50 | 5 | 25 | 3 | .430 | +.016 |
| 11 | 250 [3] | 7 | — | — | .431 | +.017 |
| 12 | 250 [3] | 10 | — | — | .441 | +.027 |

[1] On 15% moisture basis.
[2] Control flour contained 0.414% ash at 15% moisture.
[3] Conducted in 2 × 6-inch metal centrifuge tube cups.

From the foregoing it appears that the accuracy and utility of the method depend upon securing intimate contact between carbon tetrachloride and all particles in phosphated and self-rising flours. Sufficient liquid must be used to permit vigorous agitations, whereas more is uneconomical. A second carbon tetrachloride treatment is necessary to effect complete removal of added phosphate and other self-rising ingredients. Second treatments invariably removed traces of mineral sediment, but no sediment resulted in the several cases where a third treat-

ment was given. Speed and time of centrifuging are also important. Optimal centrifuging conditions for satisfactory removal of salts from a 20-gram charge have proved to be 7 minutes for the first treatment and 5 minutes for the second, at a speed of between 1000 and 1100 r.p.m.

Further studies were made to test the accuracy of the modified method when the ash content of the original flour in self-rising flour is high. Three flours having ash contents of 0.713%, 1.181%, and 1.200%, respectively, were made into self-rising flour by addition of the usual ingredients and analyzed for moisture and for ash. Duplicate samples were then treated with carbon tetrachloride for recovery of the original flour; dried and analyzed for moisture and for ash. The results are shown in Table III.

TABLE III

ASH CONTENT OF LOW-GRADE FLOURS BEFORE AND AFTER ADDITION OF SELF-RISING INGREDIENTS

| Ash Content [1] | | | |
|---|---|---|---|
| Original flour | Total as self-rising | Recovered flour | Deviation from original |
| % | % | % | % |
| | | 0.710 | |
| | | .700 | |
| 0.713 | —— | Ave. .705 | −.008 |
| | | .718 | |
| | | .720 | |
| 0.713 | 4.976 | Ave. .719 | +.006 |
| | | 1.170 | |
| | | 1.172 | |
| 1.181 | —— | Ave. 1.171 | −.010 |
| | | 1.165 | |
| | | 1.165 | |
| 1.181 | 5.037 | Ave. 1.165 | −.016 |
| | | 1.183 | |
| | | 1.179 | |
| 1.200 | —— | Ave. 1.181 | −.019 |
| | | 1.184 | |
| | | 1.190 | |
| 1.200 | 4.934 | 1.187 | −.013 |

[1] On the basis of 15% moisture.

In every case, the ash results on recovered flours agree closely, being well within the limits of experimental error. The data of Table III show that the ash of each of the three plain flours that were subjected to the carbon tetrachloride treatment was practically identical with the original ash.

### Conclusions

A modified method is described for the removal of added ingredients from phosphated and self-rising flours so that the true ash content of the original flours can be determined.

The method is rapid, accurate, and economical as to time and materials.

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## GENERAL REPORT 1933–34 COMMITTEE ON CAKE BAKING TESTS

G. L. ALEXANDER,[1] *Chairman*

(Read at the Annual Meeting, June 1934)

For the past year the Committee on Cake Baking Tests has devoted its efforts chiefly toward the standardization of formula ingredients. Studies of dried egg albumin, powdered skim-milk, and leavening agents have been made, with good progress reported.

It is recommended that further study be given to dried egg albumin before standard quality specifications are established for this material.

The work of the subcommittee on leavening indicated that no changes were in order this year. It was decided that no important variations are to be expected due to lack of uniformity of the soda or cream of tartar; because the purity of these chemicals is controlled through the specifications given in the pharmacopea.

Work done so far on dried skim-milk leads to a recommendation that the extra grade, spray dried skim-milk, be used for the cake baking test.

Suggestions for the attention of next year's Committee on the Cake Baking Test are—

The comparative merits of the beater and whip should be investigated for use in the mixing procedure of the official cake baking test.

The use of a constant Beaumé sugar solution should be worked out for Supplement A of the cake baking test. It is felt that such a solution will maintain a uniform batter consistency as the sugar is increased in the formula.

The directions for procedure in the official cake baking test should be re-written in greater detail for the benefit of those not familiar with cake making practice.

There is need of a more accurate and definite explanation of cake characteristics and scoring terms, their significance, and their value in the cake score.

[1] Commercial Milling Company, Detroit, Michigan.

# CAKE-BAKING METHOD FOR TESTING SOFT WHEAT FLOURS

## Standardization of Cream of Tartar

LAURA K. TRACK

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(Read at the Annual Meeting, June 1934)

Part of the work of standardizing the cake formula for the purposes of the basic cake-baking method of the A. A. C. C. for testing soft wheat flours lies in establishing the value of leavening, as cream of tartar and soda bicarbonate, in the formula and the effects of its variations. In order to study the effects of greater and/or lesser amounts of leavening when the basic cake formula of the A. A. C. C. (Cereal Chem. 9: 407) or any of its variations is used, several series of baking tests were outlined in which the basic formula was taken without variables as to liquid, sugar, and shortening but,

(*a*) The leavening was increased by 10% increments to approximately 4% of the flour by weight;
(*b*) With leavening at 5% or approximately the proportions used in household cake baking;
(*c*) With decrements of 5% up and including a 20% decrease under the original amount.

The above increases and decreases in quantities of leavening were used throughout a series of additional bakings in which the weight of sugar in the basic formula was increased by 10% increments up to and including an increase of 50% over the original amount.

The data resulting from a study of the effects upon cake volume of varying the proportions of leavening in the presence of increased amounts of sugar, the liquid and shortening content of the formula being kept constant, when using the A. A. C. C. basic cake formula for soft wheat flours, are shown in Table I.

These data show that with but one exception (that series of bakes in which 10% more sugar was added to the test formula) an increase in the amount of leavening over and above that originally specified for the A. A. C. C. basic cake formula resulted in a decrease in loaf volume. An increase of sugar in the A. A. C. C. formula brings about decreases in loaf volume with and without the presence of additional leavening,

TABLE I

EFFECTS UPON VOLUME OF VARYING PROPORTIONS OF LEAVENING UNDER INCREASED PROPORTIONS OF SUGAR, THE LIQUID AND SHORTENING BEING KEPT CONSTANT, WHEN USING THE A. A. C. C. BASIC CAKE FORMULA FOR TESTING SOFT WHEAT FLOURS

| Sugar | | Amount of leavening | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | | Original amount | 10% increase | 15% increase | 20% increase | Household proportions | 5% decrease | 10% decrease | 15% decrease | 20% decrease |
| | Cream of tartar | 6 g. | 6.6 g. | 6.9 g. | 7.2 g. | 8.67 g. | 5.7 g. | 5.4 g. | 5.1 g. | 4.8 g. |
| | Soda | 3 g. | 3.3 g. | 3.45 g. | 3.6 g. | 4.33 g. | 2.85 g. | 2.7 g. | 2.55 g. | 2.4 g. |
| *Grams* | | *Loaf volumes in cc.* | | | | | *Loaf volumes in cc.* | | | |
| 250 | | 874 | 869 | 848 | 874 | 889 | 853 | 850 | 851 | 778 |
| 275 | | 892 | 872 | 848 | 862 | 856 | 853 | 844 | 839 | 763 |
| 300 | | 841 | 847 | 836 | 801 | 792 | 813 | 831 | 771 | 751 |
| 325 | | 812 | 810 | 815 | 744 | 774 | 798 | 816 | 757 | 736 |
| 350 | | 795 | 727 | 744 | 710 | 728 | 758 | 798 | 749 | 710 |
| 375 | | 706 | 707 | 713 | 661 | 692 | 699 | 714 | 713 | 692 |

the gross effect becoming greater and greater as the amount of leavening is increased concurrently with an increase in the amount of sugar.

When the leavening was decreased there was a decrease in cake volume in comparison with the original or control bake in every instance. However, no additional decrease in cake volume was noted by the inclusion of 10% more sugar in the formula when the leavening was reduced 5%. In this same added-sugar-series of tests, however, a decrease of 10% in the amount of leavening produced a slight decrease in cake volume. Decreases in cake volume, comparing the original amount of sugar used to the greatest amount of sugar used were less where the smaller amounts of leavening were used.

An increase in sugar over the original 250 g. added with the cream of tartar in the last stage of mixing effected no change in volume relations from that obtained by the regular procedure, the peak volume being associated with the 10% increase in sugar. However, there was an indication that the falling off in volume with sugar increases beyond this point was more marked than when the increased amounts of sugar were included in the mix from the beginning. These observations, however, are not conclusive. When the 10% increase in sugar was dissolved in milk the volume was slightly greater than when the sugar was added in the usual way.

As increased sugar in the formula had such a marked effect on cake volume, a further study was made of this problem. In making this study the sugar content of the basic formula was increased by 10% increments, omitting the cream of tartar and soda from the formula. The results of these tests are given in Table II. The data are self-explanatory and need not be further discussed.

TABLE II

EFFECTS OBTAINED BY VARYING THE PROPORTIONS OF SUGAR, WHERE THE CHEMICAL LEAVENING AGENT IS OMITTED, USING THE A. A. C. C. BASIC CAKE FORMULA

| Ideal score | | Original proportions | Sugar increased by 10% increments | | | | |
|---|---|---|---|---|---|---|---|
| | | Flour, 260 g. | 260 g. | 260 g. | 260 g. | 260 g. | 260 g. |
| | | Sugar, 250 g. | 275 g. | 300 g. | 325 g. | 350 g. | 375 g. |
| *External* | | | | | | | |
| Symmetry | 10 | 8 | 8.5 | 9.0 | 9.5 | 10.0 | 9.0 |
| Volume | 15 | 1 (596 cc.) | 2.5 (628 cc.) | 2.5 (634 cc.) | 4.0 (666 cc.) | 5.0 (676 cc.) | 5.0 (680 cc.) |
| Crust | 5 | 4.5 | 5.0 | 5.0 | 4.0 | 3.0 | 2.5 |
| *Internal* | | | | | | | |
| Texture | 30 | 25 | 27.0 | 25.0 | 20.0 | 15.0 | 15.0 |
| Grain | 25 | 20 | 22.0 | 24.0 | 18.0 | 15.0 | 10.0 |
| Color | 15 | 14 | 15.0 | 15.0 | 13.0 | 12.0 | 11.0 |
| Total score | 100 | 72.5 | 80.0 | 80.5 | 68.5 | 60.0 | 52.5 |

A similar study was made on the shortening phases of the problem, increasing the shortening in the formula by 25% increments. The data are shown in Table III.

TABLE III

EFFECTS OBTAINED BY VARYING THE PROPORTIONS OF SHORTENING WHERE CHEMICAL LEAVENING IS OMITTED, USING THE A. A. C. C. BASIC CAKE FORMULA

| Ideal score | | Original proportions | Shortening increased by 25% increments | | | | |
|---|---|---|---|---|---|---|---|
| | | Flour, 260 g. | 260 g. | 260 g. | 260 g. | 260 g. | 260 g. |
| | | Shortening, 65 g. | 81 g. | 97 g. | 113 g. | 129 g. | 145 g. |
| *External* | | | | | | | |
| Symmetry | 10 | 8.0 | 9.0 | 9.5 | 8.5 | 8.5 | 8.5 |
| Volume | 15 | 1.5 (608 cc.) | 3.0 (643 cc.) | 4.5 (667 cc.) | 4.5 (666 cc.) | 2.0 (616 cc.) | 1.5 (613 cc.) |
| Crust | 5 | 4.0 | 4.75 | 5.0 | 5.0 | 4.0 | 3.0 |
| *Internal* | | | | | | | |
| Texture | 30 | 15.0 | 20.0 | 25.0 | 25.0 | 22.0 | 20.0 |
| Grain | 25 | 15.0 | 20.0 | 25.0 | 20.0 | 15.0 | 15.0 |
| Color | 15 | 12.0 | 13.0 | 14.0 | 14.0 | 12.0 | 12.0 |
| Total score | 100 | 65.5 | 69.75 | 83.0 | 77.0 | 63.5 | 60.0 |

Further tests were made to study the effect on cake volume of varying the proportions of sugar and shortening with and without the addition of leavening to the cake formula. The data relative to these tests are given in Table IV. From these tests it was found that with an increase in shortening the cake crust becomes tenderer, browner, crinkly; the liners fell away from the cakes. With an increase in sugar the crust becomes tougher, thicker, pitted, and browner.

With increases in shortening, up to a 100% increase, holes developed.

TABLE IV

SHOWING DIFFERENCES IN VOLUME RELATIONS WITH SUGAR OR SHORTENING VARIABLE, WITH AND WITHOUT LEAVENING

| Shortening, variable | | | Sugar, variable | | |
|---|---|---|---|---|---|
| Amount of shortening | 6 g. Cream of tartar 3 g. Soda | No leavening | Amount of sugar | 6 g. Cream of tartar 3 g. Soda | No leavening |
| *Grams* | *Loaf volume, cc.* | *Loaf volume, cc.* | *Grams* | *Loaf volume, cc.* | *Loaf volume, cc.* |
| 65 | 815 | 608 | 250 | 874 | 596 |
| 81 | 822 | 643 | 275 | 892 | 628 |
| 97 | 749 | 667 | 300 | 841 | 634 |
| 113 | 735 | 666 | 325 | 812 | 666 |
| 129 | 702 | 616 | 350 | 795 | 676 |
| 145 | 675 | 613 | 375 | 706 | 680 |
| Shortening increased, each step, 25% over original amount. | | | Sugar increased, each step, 10% over original amount. | | |

Beyond this, the grain was closer and more furry, with the holes smaller and less numerous. With an increase in sugar, up to 30% increase, the cake crumb became more feathery. From that point on the upper part of the cake became porous, the lower part, close and gummy, becoming progressively more marked with sugar increases. Cakes with sugar at 140% and 150% levels collapsed entirely.

TABLE V

A STUDY OF ADJUSTMENTS OF LEAVENING AND LIQUID WHERE SHORTENING IS VARIABLE

| Conditions | Shortening | | | | | |
|---|---|---|---|---|---|---|
| | Original amount | 25% increase | 50% increase | 75% increase | 100% increase | 125% increase |
| Cream of tartar, 6 g.<br>Soda, 3 g.<br>Milk (liquid), 230 cc. | 856 cc.<br>851 cc. | 863 cc.<br>848 cc. | 798 cc.<br>771 cc. | 749 cc.<br>725 cc. | 661 cc.<br>680 cc. | 643 cc.<br>684 cc. |
| Cream of tartar, 6 g.<br>Soda, 3 g. | 230 cc. milk,<br>851 cc. | 10% less milk,<br>851 cc. | 20% less milk,<br>815 cc. | 30% less milk,<br>828 cc. | 40% less milk,<br>819 cc. | 50% less milk,<br>783 cc. |
| Cream of tartar, 6 g.<br>Soda, 3 g. | 230 cc. milk,<br>853 cc. | 5% less milk,<br>841 cc. | 10% less milk,<br>840 cc. | 15% less milk,<br>786 cc. | 20% less milk,<br>754 cc. | 25% less milk,<br>698 cc. |
| Cream of tartar, variable<br>Soda, "<br>Liquid milk, " | | Cr. Tr., 5.5 g.<br>Soda, 2.75 g.<br>10% less milk,<br>838 cc. | Cr. Tr., 5.5 g.<br>Soda, 2.75 g.<br>20% less milk,<br>824 cc. | Cr. Tr., 5.5 g.<br>Soda, 2.75 g.<br>30% less milk,<br>816 cc. | Cr. Tr., 5.5 g.<br>Soda, 2.75 g.<br>40% less milk,<br>766 cc. | Cr. Tr., 5.5 g.<br>Soda, 2.75 g.<br>50% less milk,<br>762 cc. |
| Cream of tartar, Variable<br>Soda, "<br>Liquid milk, " | | Cr. Tr., 5.5 g.<br>Soda, 2.75 g.<br>5% less milk,<br>828 cc. | Cr. Tr., 5.5 g.<br>Soda, 2.75 g.<br>10% less milk,<br>834 cc. | Cr. Tr., 5.0 g.<br>Soda, 2.5 g.<br>15% less milk,<br>762 cc. | Cr. Tr., 5.0 g.<br>Soda, 2.5 g.<br>20% less milk,<br>710 cc. | |
| | | Cr. Tr., 5.0 g.<br>Soda, 2.5 g.<br>10% less milk,<br>831 cc. | Cr. Tr., 5.0 g.<br>Soda, 2.5 g.<br>20% less milk,<br>833 cc. | Cr. Tr., 5.0 g.<br>Soda, 2.5 g.<br>30% less milk,<br>696 cc. | Cr. Tr., 5.0 g.<br>Soda, 2.5 g.<br>40% less milk,<br>715 cc. | Cr. Tr., 5.0 g.<br>Soda, 2.0 g.<br>50% less milk,<br>715 cc. |

A study was also made relative to adjustments in the amount of leavening and liquid in the formula where the shortening is variable. The results of these tests are given in Table V. From these tests it appears that if the shortening is increased 25% the amount of milk should be reduced 10%. A further reduction in the amount of milk to 15% is desirable if the quantity of shortening is increased to 50%.

## Conclusions

In the foregoing tests with the basic cake formula for testing soft wheat flours, there was a difference in tolerance for sugar and for shortening reflected according to whether or not leavening was used.

Without leavening, the 50% increase in shortening gave optimum results in volume, grain, and texture, with a 75% increase in shortening giving the same volume results; the cake, however, falling off in symmetry and grain.

With leavening, a 25% increase in shortening gave optimum results, accompanied by a sharp falling off in volume when increases beyond that were used except where liquid adjustments downward were made, in which case the tolerance reflected was more nearly in line with that shown in tests made without leavening.

Without leavening, increasing the sugar content by 10% increments, gave a gradual increase in cake volume, the greatest increase in sugar giving the largest volume. Symmetry, texture, and grain fell off after a 50% increase.

With leavening, the optimum results were reached by a 10% increase in sugar.

Throughout these tests only one flour was used, the attempt being made to produce even volumes by means of adjustments in leavening or liquid or both so as to reflect the value of the sugar or shortening alone rather than the carrying value of the formula as a whole.

Leavening had the tendency to cut the apparent tolerance to sugar and to shortening, but adjustments in the amount used showed no appreciable results.

Adjustments of liquid showed that apparent tolerance to sugar and to shortening as reflected by volume, grain, and texture, could be increased appreciably within the limits of 75% for shortening and 20% for sugar, by reducing the amount of liquid in the formula.

It is recommended that the amounts of cream of tartar and soda as in the original formula, be kept.

# MILK POWDER FOR THE CAKE BAKING TEST [1]

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(Read at the Annual Meeting, June 1934)

The latest formula for the A. A. C. C. cake baking test, as reported at the 1933 meeting, calls for the use of 230 g. of skim-milk made up of 30 g. of milk powder dissolved in 200 cc. of water. One of the activities of the Cake Baking Test Committee this year has been the standardization of materials used in the cake baking test, and this report is intended to open a discussion on the type and grade of milk powder to be used in the test.

The work of the American Dry Milk Institute has done much to simplify the problem of milk standardization. According to the latest report entitled "The Grading of Dry Skim Milk" [2] it has been found convenient to divide dry skim-milk into three major divisions according to method of manufacture, namely, spray, vacuum drum, and roller. These types are in turn divided into three grades of quality, Extra, Standard, and Third Grade. The requirements for each grade are described in detail in the bulletin mentioned. Some changes have been made since the original publication in 1929. These are, a reduction in acidity, slight increase in solubility index (in the case of the spray and roller dried milk) and a decrease in bacteria count. Methods for testing are described in the bulletin.

Skim-milk powder that meets the requirements of Extra Grade is of high quality. It is the grade that is used for human consumption. There are many brands of milk powder that meet the specifications of this grade. Certainly any milk powder that is used in the cake baking test must first of all be of Extra Grade. It is, therefore, recommended that the milk powder used in the A. A. C. C. cake baking test conform to the requirements of the Extra Grade as defined by the American Dry Milk Institute.

The selection of the type of milk powder is the next consideration. As stated above, there are available milk powders made according to the spray, vacuum drum, and roller process. There is considerable difference in the size of the particles of the roller process compared with the other two. As pointed out by Washington Platt, in a private

[1] Subcommittee report, 1933–34 Committee on Cake Baking Tests.

[2] American Dry Milk Institute, 221 N. LaSalle Street, Chicago, Ill. "The Grading of Dry Skim Milk," 1929.

communication to the writer, for uniform results, "it would be well to specify that the milk powder be put through a sieve of definite fineness before adding to the butter, provided it is added dry." However, it has been specified in the cake baking test that the milk powder should be thoroughly dissolved in the water used in the test. This can easily be accomplished with the help of a malted milk mixer. The size of the milk powder particles is not of importance except in so far as it effects solubility.

A study was made of the effect of the three types of skim-milk powder in the standard A. A. C. C. cake baking test as outlined at the 1933 convention. The powder used conformed to the requirements of Extra Grade. The same absorption was used with each type of milk powder, although the roller process powder apparently increased the absorption. A high grade commercial cake flour was used in the test. During the mixing of the batter it was evident that the roller process milk had a greater emulsifying effect than either the spray or vacuum drum powders. This difference was carried through into the test cake and was reflected in slightly greater volume and better symmetry. The cakes were scored by the official A. A. C. C. method. The results are given in Table I. Skovholt and Bailey [3] noticed differences in

TABLE I

COMPARISON OF SPRAY, VACUUM DRUM, AND ROLLER PROCESS MILK POWDER ON THE QUALITY OF TEST CAKES

| | Perfect score | Spray | Vacuum drum | Roller |
|---|---|---|---|---|
| Symmetry | 10 | 9.5 | 9.2 | 9.6 |
| Volume | 15 | 14.5 | 14.0 | 15.0 |
| Crust | 5 | 4.5 | 4.5 | 5.0 |
| Texture | 30 | 27.0 | 27.0 | 28.0 |
| Grain | 25 | 23.0 | 23.0 | 23.0 |
| Color | 15 | 15.0 | 15.0 | 15.0 |
| Total | | 93.5 | 92.7 | 95.6 |

behavior of the spray and roller process milk powders in bread doughs. The latter "caused the doughs to reach their maximum plasticity with less mixing than when 'spray' milks were used." The detailed score of the cakes is given in Table I. The total score of 95.6 for the roller process compared to 93.5 for the spray process, as judged by the writer. This would indicate that roller process milk has an advantage over the spray powder. However, the greater solubility and greater uniformity, in the experience of those qualified to judge, which is pos-

[3] Skovholt, Oscar, and Bailey, C. H. Relation of quality in dry skim milk to baking strength. Cereal Chem. 8: 374–380 (1931).

sessed by the spray powders, recommends its adoption. The A. A. C. C. cake test is designed to show up differences in cake flours by use of proper supplementary tests. Persons using the test must be satisfied their results are dependable. The selection of spray process milk powder, Grade A, makes available a high quality milk powder.

The relatively higher insolubility of the roller process milk is due to the higher drying temperature which coagulates some of the albumin and casein and precipitates some of the mineral salts.

It was the intention to make the formalin titration of the protein materials in reconstituted milk from the three types of milk powders and from several brands of each type, but the stress of business did not permit the carrying out of these plans. There is a possibility that this titration will assist in the selection of milk powder of proper physical condition.

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## DIASTATIC SUPPLEMENTS FOR THE A. A. C. C. BAKING TEST. II

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(Read at the Annual Meeting, June, 1934)

The desirability of eliminating diastatic activity as a variable factor in test baking has been thoroughly discussed elsewhere (Jørgensen (1931), Blish and Hughes (1932), Elion (1932), Frey (1933) ). Last year this subcommittee (Landis and Frey, 1933) report was concerned with the development of an equation whereby the sugar level in the dough could be predetermined from the diastatic values of the flour:

$$S_a = 2.1(t - 0.6) = \frac{s}{0.301} \log t - p$$

$S_a$ being the added sugar in percent, $t$ time in hours, and $p$ and $s$ the primary and secondary saccharogenic values respectively (Landis, 1934).

In order to eliminate the effect of the diastatic factor on the final results as completely as possible it seems desirable to attempt to maintain a constant sugar concentration in the test loaf. Morison (1925) finds the residual sugar content of a large number of commercial loaves to vary from 2.79% to 5.81%. Taking for the present 4% as an adequate value and noting that $t = 3.9$ hours for the Standard Baking test conditions, the above equation becomes:

$$S_a = 10.93 - (1.96s + p)\ ^{1}$$

[1] For convenience in rapid calculation these constants may be rounded off to $S_a = 11 - (2S + p)$.

This does not take into consideration the enzymic relationships during oven spring. The diastatic rate has diminished to a relatively small value at this point, and may, for the present, in comparison with the fermentation rate, be generally neglected. Assuming an average temperature of 40° C. in the loaf during the first 6 minutes in the oven (the fermentation rate becomes negligible slightly above 50° C.) and a temperature coefficient of 2 for each 10° rise, it may be estimated that the amount of sugar fermented during oven spring is approximately 0.42%, say 0.5% average over a variety of conditions. Hence, we must increase the value of $S_a$ calculated for the end of proof time by 0.5%, *i.e.*,

$$S_a = 11.4 - (1.96s + p)$$

In order to test the reliability of this equation, a series of ten flours with Rumsey values varying between 80 to over 400, from several major wheat growing parts of the United States, were tested. The diastatic values were determined (cf. Landis, 1934) on each flour, $S_a$ calculated and the flours baked by the "short method" proposed by Merritt, Blish, and Sandstedt (1932), using variable absorption and sugar as calculated. The finished loaves were then analyzed for fermentable sugars. The results are given in Table I.

TABLE I

THE RELIABILITY OF SUGAR LEVEL CALCULATIONS

| Flour | Saccharogenic values | | | | $S_r$ (Basic) (Calculated) |
|---|---|---|---|---|---|
| | $p$ | $s$ | $S_a$ | $S_r$ | |
| Northwestern Patent | 4.52 | .83 | 5.3 | 3.7 | 1.7 |
| Southwestern Patent | 2.50 | .57 | 7.8 | 3.3 | −0.8 |
| Kansas Hard Winter | 2.68 | .43 | 7.9 | 3.5 | −0.9 |
| Montana Marquis | 7.00 | 1.32 | 1.7 | 3.0 | 5.3 |
| Washington Baart | 2.62 | 1.03 | 6.8 | 4.3 | 0.2 |
| Dicklow | 2.42 | .81 | 7.4 | 4.1 | −0.4 |
| Idaho Hard Winter | 3.20 | 1.21 | 5.8 | 4.2 | 1.2 |
| Western Club | 3.52 | .97 | 6.0 | 3.4 | 1.0 |
| Idaho Turkey | 3.20 | .79 | 6.7 | 4.4 | 0.3 |
| Western Patent | 3.52 | 1.48 | 5.0 | 3.9 | 2.0 |
| Mean value | | | | 3.78 | |
| Standard deviation | | | | 0.48 | |
| Coefficient of variation | | | | 12.7 | |

The mean value 3.78 is nearly ¼% lower than the calculated value. This is to be expected, since the diastatic values were determined in fermenting suspensions which have been shown to give a higher sugar level than doughs. The greatest negative deviation found was in the case of the high diastatic Montana Marquis. This is probably due

to the greater error in the determination of such high diastatic values, as well as to the fact that the difference between suspensions and doughs is more marked in such a case.

The theoretical values to be expected in the basic procedure (2.5% sugar) indicate the marked effects which lack of control of the diastatic factor may be expected to introduce into a series of tests in which a constant diastatic supplement is used.

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## BOOK REVIEWS

**Les cereales.** By R. Legendre, Directeur du Laboratoire de Physiologie comparée à l'École des Hautes Études. Published by Librairie Armand Colin, 103 Boulevard Saint-Martin, Paris. 1935. 218 pages. Price 10.5 francs.

This appears to be one of a series of semi-technical texts published under the general editorship of Prof. Paul Montel (Sorbourne) who uses the slogan "Popularization without lowered standards" to describe this effort. Legendre's contribution covers a wide scope of subject matter. It includes the following chapters: botany, anatomy and chemical composition of the grains, biology of the grains, economic importance of the cereals, statistics of production, damaged cereals (pests), means of utilization and conservation. Accordingly we find a rather inclusive treatment of the position of cereals, chemical, technological and economic in the family of foods.

Legendre's physiological training becomes immediately apparent in his treatment of this broad subject. In general he has selected his principal technical and scientific facts from responsible sources. Thus he has used the recently announced concepts of the constitution of carbohydrates in developing his treatment of that phase of the chemical composition of the cereals; in fact this section of the book is expanded somewhat out of proportion to its practical significance.

The discussion of the biology of the cereals appears to be sound and well balanced, with suitable emphasis upon several biochemical aspects of this phase of the subject. In the next chapter appropriate emphasis is laid upon the importance of cereals in human dietaries. The author also stresses the role that improved transportation facilities have played in facilitating the manufacture of superior flour of constant properties and composition. Here, and again in the closing chapter, Legendre has traced in an attractive manner certain historical episodes that contributed to the conservation and effective utilization of grain.

Thus for an exceedingly modest sum, as judged by prices now charged for new technical publications, particularly from the German publishing houses, one can acquire a useful monograph which should be of service not only to the novice or casual reader, but to the specialist as well.

C. H. BAILEY.

**The Law of Patents for Chemists.** Second Edition. By Joseph Rossman, Editor of Journal of Patent Office Society. Published by Williams & Wilkins Co., Mt. Royal and Guilford Aves., Baltimore, Md. 1934. 378 pp., 8 x 5¾ in.; cloth. Price $4.50.

Demand for the first (1932) edition of this book proved the need by quickly exhausting the available supply. To bring the contents of subsequent printings up-to-date and cover new phases of chemical inventions the author has revised the contents of the original twenty chapters through the addition of new material, by rewriting original text to conform to new interpretation of patent law, and by substituting more explicit examples to illustrate workings of principles involved, and has included an additional chapter to cover special problems found in medical patents, inventions in which living organisms, such as bacteria, yeasts, and fungi, and their enzymes are used, patents based upon formulas or recipes for foods, and metallurgical inventions.

In this review more attention is given to the new subject matter than to the contents of the book as a whole, which to a large extent is covered in the review of the first edition published in CEREAL CHEMISTRY, May 1932, p. 328.

Inclusion of 75 additional pages in this second edition does not tell the whole story. Use of a whiter and better quality of paper gives easier reading of the text even though the type-face is smaller and more words appear on each page.

Typical of new subject matter added to original chapters are: discussion in Chapter III on university and technological institute patent policies; statement in Chapter IV, citing how an invention can be legally protected through secrecy; explanation in Chapter VII why the use of borax solution to prevent blue mold

decay in oranges is not a patentable "manufacture"; listing in Chapter IX of steps in inventing followed by inventors and subsequent discussion with clarifying examples on several points of question in what constitutes an invention; correction in Chapter X, specifying that an applicant for a patent must believe himself to be the original and first inventor when he completes his invention instead of when he files his application on an invention in the United States; caution in Chapter XII relative to the use of trade-names to define materials used, to the preparation of drawings and photomicrographs, and to the danger of oversimplification of describing invention in patent applications; clearing up in Chapter XIII of disputed points regarding what shall be covered in claims made for an invention; statement of policy in Chapter XIV lately announced by the Commissioner of Patents in regard to species and generic inventions that can be claimed in an application, also the bases upon which an application for patent may be made "special" are given; clarifying phrases, clauses, and sentences in Chapter XVI on the subject of interferences defining conception and reduction of an invention to practice, preservation of evidence, and establishment of priority; treatise in Chapter XIX on employer-employee patent relations concerning inventorship, invention ownership, government employee patents, and shop rights; case citations in Chapter XX covering enforcement of patent rights in infringement cases and exclusion of foreign products which infringe domestic rights, and new miscellaneous information in Chapter XXI, chief of which is the rewritten section on The Appraisal of a Chemical Invention by D. B. Keyes, Head of the Chemical Engineering Division, University of Illinois, also to be published as a portion of Prof. Keyes' book on research methods.

Special Chemical Patent Problems constitutes the subject of Chapter XV, the wholly new chapter in this second edition. Chemists engaged in food, particularly cereal food, research should be especially interested in this chapter because of decisions made by the courts relative to the patentability of cereal breakfast foods and vitamin preparations.

E. S. STATELER.

**Starch and Flour Quality.** By C. L. Alsberg. Wheat studies of the Food Research Institute, Vol. 11, No. 6, pages 229–254. February, 1935. Price 50¢.

In this study existing information bearing on the influence of wheat starch on the baking value of flour has been collected, some of the gaps in the knowledge relative to this substance pointed out, and some problems awaiting solution presented. A discussion of those properties of wheat starches which have a bearing on baking quality is presented in detail.

It is pointed out that starch absorbs about as much water as all other flour constituents combined and that, therefore, the water absorption of flours might depend to some extent upon the water capacity of their starches. The susceptibility of starch to diastase is one of the factors affecting the diastatic power of flour and in consequence panary fermentation. This susceptibility to attack depends not only upon the size of the starch granules and upon their intrinsic ability to resist conversion into sugar by diastase, but also upon their location—whether surrounded by a protecting envelope of gluten or freed from it. It is because fine grinding separates large numbers of granules from the gluten envelope that fine grinding increases the apparant diastatic power of flour. But it also injures starch granules so that they swell in cool water, thereby increasing the absorption of the flour; and at the same time some starch goes into solution. In consequence, diastatic power is increased because injured granules and dissolved starch are readily attacked by the enzyme. Finally, changes that the modified starch of bread undergoes in time seem to be the major factor in causing bread to grow stale.

D. A. COLEMAN.